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AMERICAN
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VOLUME 25 DECEMBER 14, 1936 NUMBER 25

Processing of

Knitted Wool Underwear Fabric

F. P. W. WILSOME

WOOL underwear has the advantage over cotton in being a better heat-retaining fabric and in having higher perspiration absorptive properties. It is true that cotton fabric can be 'raised' so that it has a hairy surface and in this manner be made soft and a better non-conductor of heat, but in spite of this the all-wool fabric is to be preferred.

Underwear must readily accommodate the movements of the human body and for this reason it is better made from a knitted than a woven fabric. So far, no modification of the weaving process has resulted in the production of a fabric having that elasticity and power to retain its shape which characterizes a suitably knitted material. It is therefore to be anticipated that for underwear purposes the knitted garment will be supreme for many years yet to come.

Since underwear comes in intimate contact with the human skin it is essential that the wool fabric used for this purpose be soft and incapable of causing irritation by rubbing harshly. Not only is it desirable to use the best quality wool for underwear garments but the finisher must have the ability to process it in such a manner as to bring out or increase its softness to the utmost extent. Harshness must be avoided at all costs.

A further point in connection with wool underwear fabric is that it must have a good color. While much of this fabric is bleached to a good white it must be recognized that a large amount is only partly bleached so as to resemble a so-called 'natural' shade. This natural shade is very slightly yellow with a tinge of pink or grey; it should be retained by the fabric after repeated washing and should never turn to a dingy or dirty tone which would, in fact, make the garment look as though it had been very carelessly washed. Sometimes this required appearance is obtained by spinning the yarn used in the fabric from wool containing a proportion of suitably dyed wool, and in this case the finisher has to avoid destroying this color in his processing.

Finally, a good quality wool underwear fabric should be unshrinkable. It is, of course, one of the disadvantages of wool that it readily felts in washing, so that the fibers become matted together. The fabric then loses much of its elasticity and acquires a harshness which takes away much of its value for underwear purposes. Different qualities of wool felt to varying degrees, but unfortunately the best qualities felt most easily and it is precisely these qualities which are employed for underwear. The finisher of underwear fabric is therefore generally called upon to chlorinate the material so that its felting properties are much reduced. This complicates the finishing process in so far as this treatment can adversely affect the color and handle of the fabric.

In this article it is proposed to describe the essential points in the processing of an underwear wool fabric from the point at which it leaves the knitting machine up to the time at which it is suitable for cutting-up and making into vests, etc.

As the fabric, generally in tubular form, leaves the knitting machine it will most probably contain lubricating oil both throughout its texture and also as splashes here and there. Also the fabric will contain a fair amount of dirt collected from the knitting machine. The first operation will thus consist of a good scouring.

It may be that the fabric has been kept in stock for some time before processing and in this case the oil stains may have become oxidized and so become much more difficult to remove. For this reason it is often the best policy to look over the fabric before scouring, and sponging with an organic solvent any oil stain which appears to be particularly dirty or noticeable. An emulsion of benzine and soap is effective for this purpose since it makes more easily removable in scouring the hardened oxidized oil stains to which it may be applied. This hand treatment may appear to be wasteful of labor but it often is very profitable in so far as it prevents trouble at later stages.

The fabric is now ready for scouring and this is an important process. Much depends on how much milling the fabric is required to have in its finished condition. As is well known, wool fibers readily felt together when pressed or rubbed together in a soapy liquor, and by working the underwear fabric during the scouring it is

possible to make it felt and so have a more compact character. In some cases it is essential that the fabric be felted or milled slightly so as to give to the fabric a full appearance but in other cases milling must be rigidly avoided. For ladies' underwear, milling is generally not required, but for men's vests a slight mill is desirable, since fabric used for this purpose is usually wanted to be somewhat thick in handle. The disadvantage of milling is that although it closes up the threads and makes the fabric fuller in handle it also reduces the elasticity or extensibility so that the fabric does not so readily accommodate body movements. It is often this unwanted loss of elasticity which decides against milling.

Scouring will be most conveniently carried out in a fulling mill (Fig. 1). Here the fabric F, usually four or five pieces sewn end to end to form a continuous band, is continuously drawn up out of a soapy liquor by a pair of wooden nipping rollers A and B and then falls back into the liquor again to await successive passages through the rollers At the back of the rollers is a so-called *spout* S having a hinged lid L provided with a weighting device W; this allows the fabric to be retarded in its ejection from the rollers so that it is subject to compression. The arrangement of this device shown in Fig. 1, has the effect of causing the fabric to felt and become more compact. If no milling is required then the fabric is allowed to pass through the spout without hindrance but if milling is required then the appropriate weighting is employed. The passage of the fabric through the soapy liquor in the trough T of the machine cleans it and constitutes the scouring process.

Some wool fabrics mill so easily that it is advisable not to use such a machine but simply to soak them in a warm soapy liquor for about one hour with an occasional stir-up with a pole. The fabric is then washed free from soap and impurities by means of warm water.

It is found that the scouring process is quite satisfactory if a solution of soap and ammonia is employed. In recent years the use of sulfonated fatty alcohols has been much recommended for wool scouring in preference to soap. But these soap substitutes lack the 'body' of soap itself and in most cases it is better to use them as an addition to the soap scouring liquor rather than as a complete substitute. There is a probability that the higher alkalinity of the soap liquor makes it more effective in scouring than a simple solution of a sulphonated fatty alcohol which is around the neutral point.

Following scouring the fabric will require a chlorination treatment so as to make it unshrinkable; most underwear wool fabrics have this treatment today since it is almost essential that the garments made from them be guaranteed unshrinkable. The process depends on the fact, discovered many years ago, that when wool is steeped for a short while in an acid solution of sodium hypochlorite it loses its power to felt or mill. At the same time, this action is accompanied by a slight impoverishment of the wool itself so as to impair its durability.

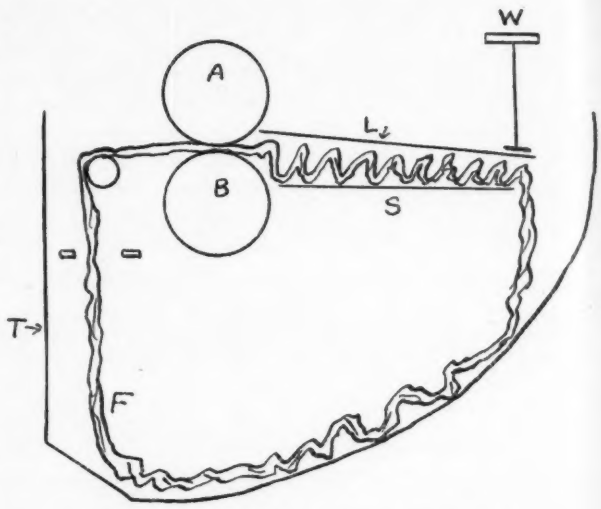

Fig. 1—Scouring and Milling Machine

In large scale practice it is the aim of the finisher to carry the treatment up to a point at which the fabric has lost most of its tendency to felt in washing whilst only a reasonable amount of impoverishment has taken place. A considerable amount of importance is thus attached to the judgment of the finisher.

It seems to be a fact that by simple chlorination of wool it is impossible to secure complete removal of the felting tendency of wool without appreciable impoverishment. If the treatment is carried up to the point at which the wool has a zero shrinkage in subsequent washing then the wool definitely suffers attack. This attack is evident in various ways, for example, the color of the wool becomes yellower and especially when the wool is washed several times. Also the wool acquires a very slimy handle when placed in a dilute solution of ammonia or other weak alkali. Its handle becomes firmer and the fabric does not wear well. If, on the other hand, the treatment is carried just too far then the wool behaves entirely unsatisfactorily. Not only does it shrink but in washing it becomes thinner and increases in size due to a break down of the power of the fabric to retain its shape. On the whole therefore, the chlorination treatment must be stopped short of that degree necessary to give zero shrinkage and as a result most so-called unshrinkable wool underwear fabric is actually shrinkable on washing repeatedly.

Within recent years attempts have been made to improve the unshrinkable treatment so as to secure absolute unshrinkability. The Wool Industries Research Association of Leeds investigated the use of chlorine gas instead of a solution of hypochlorite and although a big improvement was at first foreshadowed very little has recently been heard of this process. In any case it would appear that the impoverishing action of the unshrinkable treat-

(Continued on page 712)

PROCEEDINGS OF THE
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DECEMBER 14, 1936

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Sixteenth Annual Meeting

THE Sixteenth Annual Meeting, held the week-end of December 4th and 5th in Providence. R. I., proved to be one of the most successful ever held, both in point of attendance and quality of program. The Council and Research Committee meetings were held Friday afternoon and many members availed themselves of this opportunity for sight-seeing trips which proved of much interest. A buffet supper was served Friday evening following which President Cady declared the meeting officially opened. Then followed a technical paper, educational motion pictures and a program of entertainment.

Saturday morning and afternoon, according to usual custom, were given over to the presentation of technical papers and the discussions which followed. The annual business meeting was held at noon. The convention drew to a close Saturday evening with the annual banquet and brief addresses by men of prominence in the Association and other lines of endeavor.

ALLISON R. FLETCHER
General Chairman
Arrangements

The various committees who have spent the last several months in the completion of details for this meeting are to be congratulated upon the excellence of the program which was prepared.

A complete stenographic report of papers presented, discussions and meetings will appear in the Proceedings of the Association in future issues of the REPORTER.

The Determination of Alkalinities and Acidities in Sulfonated (Sulfated) Oils

Committee Report No. 6

RALPH HART*

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JULIUS JAREK.
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E. SEGESSEMAN, *National Oil Products Co.*

THE most important polar groups in sulfonated oils are: (a) the neutralized organically combined sulfuric anhydride, (b) the neutralized carboxyl group, or soap, and (c) the free carboxyl group or free fatty acids. As examples of the practical importance of these groups, the following may be cited. The quality of the finished sulfonated oil may be controlled to a considerable extent during manufacture by regulating the formation of free fatty acids, which are influenced particularly by the composition, concentration, pH, and temperature of the wash-liquors as well as the manipulations during the washing process. Again, the stability of the sulfonated oil itself depends a good deal upon the amount of soap it contains. A product containing a small amount of soap, i.e., low in alkalinity, may turn turbid, liberate sulfuric acid upon heating or storage, and ultimately separate salt solution; it is furthermore less stable to salt solutions, although more resistant to hard water. On the other hand, when highly neutralized, the miscibility of sulfonated oils with raw oils is greatly impaired although the emulsifying properties may be enhanced. Moreover, in the application of sulfonated oils, the requirements as to acidity and alkalinity differ widely; thus, in the finishing of fabrics or for soaking of raw yarns, a minimum of polar groups and a maximum of raw oils are indicated; whereas in dyeing, wetting-out, dispersion of dyestuffs, and scouring a neutralized product or one adjusted to a certain pH is preferred.

In this report the subject is discussed in the following order: methods for the determination of (1) alkalinity, (2) acidity and free alkali, (3) free-and-combined fatty acids, (4) special methods for samples containing interfering substances, (5) committee results and comments, and (6) detailed procedures for methods proposed as standards.

TOTAL ALKALINITY

Wizoeff Method—The Wizoeff[14] method fo alkalinity in sulfonated oils is as follows: 5 grams of the sample is dissolved in 95cc of water and titrated with half-normal hydrochloric or sulfuric acid until neutral to phenolphthalein. Some work carried out in the writer's laboratory indicates that solutions of neutralized sulfonated castor or olive oil are practically neutral to phenolphthalein (color change about pH 8.3). Hence, the Wizoeff method does not determine the alkali bound as soap, but only the excess alkali. The low pH of neutralized sulfonated oils undoubtedly accounts for their ineffectiveness in degumming raw silk, which requires degumming agents with a pH above 9.0.

A.O.C.S. Method—The standard method of the American Oil Chemists' Society[12] for alkali combined as soap is to acidify the sample dissolved in water and determine the acidity of the extracted fatty acids. Obviously, this method is only applicable to sulfonated oils where the amount of free fatty acids is nil or known.

Bureau of Standards Method—Alkali combined as soap in sulfonated oils is conveniently estimated, in the absence of interfering substances, by titrating with mineral acids in the presence of methyl orange. The determination is based upon the well-known facts that mineral acids combine with the alkali of the soap (liberating free fatty acids) and that the mixture remains alkaline to methyl orange (color change pH 2.9 to 4.0) until the soap is quantitatively decomposed. This is the method given in Commercial Standard CS43-32[1], which is similar to the official

*Chairman, Sub-Committee on Sulfonated Oils.

method of the American Leather Chemists' Association[10]. The procedure is as follows:

> Weigh 10 grams of the oil into a 250 ml. Erlenmeyer flask and dissolve in 150 ml. of water, warming to obtain solution if necessary. Add 30 grams of granulated sodium chloride, 25 ml. of ether, and 5 ml. of methyl orange (0.1 per cent solution) and titrate with approximately 0.5 N sulfuric acid.

This committee has already shown[6] that 5 drops of the indicator gives a more reliable end-point than the larger amount. This method is recommended by the committee with certain changes, namely, proper amount of indicator and more details in reaching the end point.

Active Ingredients Method—Another method for total alkalinity corresponding to soap and sodium carbonate, which is applicable in the presence of sodium acetate, is the procedure for "Alkalinity in Presence of Sodium Carbonate," described in Committee[7] Report No. 3. According to this method, a water solution of the sample is acidified with a known quantity of mineral acid, the active ingredients extracted, and the titration of the water layer completed, using phenolphthalein as the indicator. The net amount of acid absorbed in the titration corresponds to the total alkalinity. The result does not include the alkalinity of the acetate.

Ash Method—Since sulfonated oils are miscible within certain limits with solutions of alkali metal salts, the alkalinity determined by the Bureau of Standards, method may not be due to soap alone, but also to alkaline salts, such as the carbonate, borate, acetate, tartarate, oxalate, etc. of sodium potassium, ammonium, or organic amines. In the presence of alkali-reacting salts whose water solutions may be titrated to a sharp end point with methyl orange as the indicator (e.g., carbonates or borates), the total alkalinity may still be determined by the above method. With salts, however, that react alkaline but cannot be titrated quantitatively, such as acetates, oxalates, etc., there seems to be no simple way of determining the total alkalinity. In such cases, the following new method is suggested which gives the combined alkalinities of the soaps and of such salts which upon ashing of the original sample are converted into alkali carbonates, borates, or phosphates.

According to this method, the sample is ashed and the alkalinity of the ash is determined, using methyl orange as the indicator. Upon ignition, soaps and alkaline salts are converted into carbonates or other titratable salts and the organically combined SO_3 is converted into sodium sulfate, thus:

$$\begin{matrix} R - SO_4Na \\ \diagdown \\ \quad COONa \end{matrix} \longrightarrow Na_2SO_4 + \text{etc.}$$

This reaction is quantitative provided the number of COONá groups is equal to or greater than the number of SO_4Ná groups. It will be noticed from the reaction that one mole equivalent of organically combined SO_3 reacts with one mole equivalent of COONá, thereby neutralizing one mole equivalent of alkali in the form of soap. It therefore follows that if the organically combined SO_3 is known, the amount of alkali lost upon ashing of the sample can be calculated. This loss in alkali, as mgm KOH per gram of sample, is given by the following formula:

(1) Alkali-loss, mgm KOH/g $= \frac{56.1}{80}$ (% combined SO_3) X 10
$= 7.013$ X % combined SO_3

Evidently, the alkalinity of the ash represents the original alkalinity of the sample minus the loss in alkalinity due to the combined SO_3, or,

(2) Total alkalinity of ash/weight of sample = fixed alkalinity of sample) minus (alkali-loss).

or,

(3) Fixed alkalinity of sample, mg. KOH/g.
= Total alkalinity of ash/weight of sample plus (alkali-loss).
= Total alkalinity of ash/weight of sample plus (7.013 × % combined SO_3).

If alkali is added to the sample before ashing, in order that there may be enough to take care of the $NaSO_4$ groups, the formula becomes:

(4) Fixed alkalinity of sample, mg. KOH/g.
= (total alkalinity of ash) minus (total alkalinity added)

weight of sample
plus (7.013 X % combined SO_3

In these formulas, the alkalinity added and the alkalinity of the ash are expressed in mgms. KOH. In the application of this method. the sample is heated for about 15 minutes with a known amount of caustic soda and an equal amount of alcohol; it is then evaporated, ashed, and the alkalinity of the ash determined.

Upon examining formulas (3) or (4), it is evident that if the alkalinities of the original sample and of the ash are known, the organically combined SO_3 may be calculated. A method for combined SO_3 based on this principle[4] has been published, which method yields results that compare favorably with those obtained by other methods.

These formulas, from the alkalinity angle, have been checked in the writer's laboratory many times and found to be reliable. A sample of sulfonated castor oil of known composition, including 2.96 per cent of sodium acetate, was analyzed for alkalinities by the ash method with the following results:

Data:	*Present*	*Found*
(a) Free-and-combined fatty acids, mg. KOH/g	36.3	36.2
(b) Free fatty acids, m. KOH/g	18.8	19.6
(c) Organically combined SO_3, per cent	2.38	2.41
(d) Alkalinity of ash, mg. KOH/g of sample	...	20.7

Results:	*Present*	*Found*
(e) Fixed alkalinity as soap, mg. KOH/g [(a) minus (b)]	16.5	16.6
(f) Fixed alkalinity of sample as soap and as acetate, mg. KOH/g [Formula (3)]	36.8	37.6
(g) Fixed alkalinity as acetate mg. KOH/g [(f) minus (e)]	20.3	21.0
(g) Sodium acetate, per cent	2.96	3.07

With the exception of the alkalinity of the ash, the rest of the data is essential for the complete analysis of a sulfonated oil. In determining the free-and-alkali-combined fatty acids in the presence of acetates (described later), prolonged washing of the ether layer with liberal portions of salt solution is required in order to wash it free from acetic acid. The washing may be considered complete when a drop or two of N/2 alkali added to the wash water reacts alkaline to phenolphthalein. Ammonium soap or free ammonia does not interfere with the method. Ammonium sulfate or chloride, however, may react in part with the acetate upon ashing, giving sodium sulfate or chloride, thereby reducing the alkalinity of the ash, which loss can not be determined.

Oleic Acid Method—An alternative method for determining total alkalinity, including the alkalinity due to sodium acetate, is the method for alkalinity in the presence of acetates given in Committee[7] Report No. 3. According to this method, the sample is heated with excess oleic acid until practically all of the interfering acetic acid is expelled. The sample is then acidified, extracted, and titrated as in the "Active Ingredients Method," except that the water layer is repeatedly extracted with ether until the latter is practically free of acetic acid and that methyl orange is used as the indicator.

Alkalinity as Ammonia—The alkalinity of ammonia soap may be determined by the Bureau of Standards' method for alkalinity. If both sodium and ammonium soaps are present and it is required to estimate the alkalinity due to each, either the ammonia or the fixed alkalinity is first determined and by difference from the total alkalinity, the other alkalinity is found. The ammonia-alkalinity may be determined by distilling the sample dissolved in water with excess caustic soda and absorbing the ammonia that distills over in a solution of standardized acid, the loss of which corresponds to the ammonia in the sample.

A more convenient method (in the absence of triethanolamine soaps), recommended by the writer[3] a number of years ago, is to determine the fixed alkalinity first. The sample is boiled with a definite quantity of standardized caustic alkali until all of the ammonia is expelled, and the alkalinity of the boiled solution is then determined. The latter represents the alkali added plus the fixed alkalinity, from which data together with the total alkalinity of the original sample, the fixed alkalinity and alkalinity as ammonia, respectively, may readily be calculated. To convert ammonia from its equivalent of mgm KOH per gram of sample into per cent NH_3, the following formula is used:

$$\% \ NH_3 = \frac{1}{10} \times \frac{17}{56.1} \text{ times (ammonia as mg. KOH/g)}$$
$$= 0.0303 \text{ times (ammonia as mg. KOH/g)}$$

Kertsz[9] and Sors[13] recommend a similar method for ammonia. A still simpler method for estimating separately the fixed and volatile alkali is given later under Free-and-Alkali-Combined Fatty Acids.

ACIDITY, FREE FATTY ACIDS, AND FREE ALKALI

According to the Wizoeff[14] method, the determination of acidity corresponding to the free fatty acids is carried out exactly as in the Wizoeff method for alkalinity, except that the titration is made with N/2 caustic solution instead of acid. Some work carried out in the writer's laboratory with sulfonated castor and olive oils indicates that the results by this method compare favorably with those obtained in alcoholic solutions, although the end point is not quite so sharp. Thus, the acidity of a sample of sulfonated castor oil was determined in a water solution and found to be 26.1 mg. KOH per gram compared with 26.5, determined in an alcoholic solution. Since the pH color-change for phenolphthalein is about 8.3, these results indicate, as already mentioned, that the completely neutralized sulfonated oil in water solutions has a pH of about 8.3, compared with about 10.2 for ordinary soaps; they also indicate that the hydrolysis of the completely neutralized sulfonated oil in water is very small. It is to be noted that the results of acidity by the Wizoeff method are unreliable in the presence of ammonia soaps, since the latter in water solutions, although acid to phenolphthalein, cannot be titrated quantitatively as is the case in alcoholic solutions.

Free fatty acids in alcoholic solution react acid to phenolphthalein until quantitatively neutralized with a strong base. It is essential, however, that the solution shall contain at least 50 per cent of alcohol at the end of the titration, otherwise hydrolysis of the soap occurs, interfering with the end-point. This requirement may not be so important with sulfonated oils as with ordinary fats. The following is the official method of the American Association of Agricultural Chemists:[11]

> Weigh 20 g. of fat, or oil, into a flask; add 50 cc. of 95 per cent alcohol that has been neutralized with dilute NaOH solution, using phenolphthalein indicator; and heat to boiling. Shake the flask thoroughly in order to dissolve the free fatty acids as completely as possible. Titrate with 0.1 N NaOH or KOH, until the pink color persists after vigorous shaking.

The committee endorses this method with the recommendations, however, that the heating be omitted and a

fat-solvent be added instead. The brine-method for free fatty acids suggested by Mr. Pingree may prove very useful, particularly in the case of oils that may be so dark as to mask the end point when titrated by the usual method. According to this method, the sample is dissolved in water, salted out by means of sodium chloride, alcohol and ether added, and the contents titrated with alkali in the presence of phenolphthalein, while shaking vigorously, until a faint pink color appears in the brine solution. Obviously the free-and-alkali-combined fatty acids may also be estimated by this method, namely, by determining first the alkalinity in the presence of methyl orange, then adding phenolphthalein and alcohol, and finally titrating for total acidity—the latter result representing the free-and-alkali-combined fatty acids. It is also obvious that the difference between the two titrations gives the acidity of the free fatty acids in the original sample. A somewhat similar method has been suggested by Fedchanko and Kashin.[2]

Since ammonium or triethanolamine soaps in alcohol solution react acid to phenolphthalein and furthermore may be titrated quantitatively with caustic alkalies, the acidity determined in alcoholic solutions in the presence of such soaps represents the sum of the free fatty acids plus the fatty acids combined with the weak bases as soap. To determine in such cases the acidity corresponding to the free fatty acids alone, the method described under Free-and-Alkali-Combined Fatty Acids should be used.

To determine free caustic alkali, the test is conducted the same as for acidity except that the titration is made with acid instead of alkali.

FREE-AND-ALKALI-COMBINED FATTY ACIDS

The determination of free-and-alkali-combined fatty acids is a convenient means, and in the presence of triethanolamine soaps the only means (excluding the ash-method for fixed alkalinity), of determining separately the alkalinity corresponding to combined fatty acids and the acidity due to free fatty acids in samples containing alkaline salts and sodium, and/or ammonium or triethanolamine soaps or salts. For example, in the presence of alkaline salts, the alkalinity combined as soap alone is given either by the free-and-alkali-combined fatty acids (in case the fatty acids are completely neutralized) or the latter minus the free fatty acids. Again, in the presence of ammonium or triethanolamine soaps, which in alcohol solutions react as free fatty acids to phenolphthalein, the acidity corresponding to the free fatty acids is given by the difference between the free-and-alkali-combined fatty acids and total alkalinity. It is also obvious that given the acidity of the free fatty acids in a sample, the ammonia combined as soap is readily determined, since it is equal to the acidity of the original sample minus the acidity of the free fatty acids.

To determine the free-and-alkali-combined fatty acids, the total active ingredients are extracted according to the A.A.T.C.C. method[6], and the free fatty acids in the extract determined in the usual way by titration in alcoholic solution. In the absence of ammonia, triethanolamine, alkaline and acid salts, or other interfering compounds, the free-and-alkali-combined fatty acids are equal to the sum of the free fatty acids and total alkalinity.

SULFONATED OILS CONTAINING INTERFERING COMPOUNDS

Samples containing ammonium salts, with or without alkaline salts, sometimes require considerable ingenuity in their analysis in order to obtain a true picture of the distribution of the alkalinities and acidities. The following scheme of analysis is suggested for the determination of alkalinities and acidities in some samples of sulfonated oils containing compounds interfering with the regular procedures—the latter refer to the methods given in detail at the end of this report. In the presence of—

Sodium and Ammonium as Soaps—Determine (a) total alkalinity by the titration method, (b) total acidity, and (c) free-and-alkali-combined fatty acids, from which may be calculated the following:

Free fatty acids = (c) minus (a) = (d)
Ammonia as soap = (b) minus (d) = (f)
Fixed alkalinity as soap = (a) minus (f)

Sodium, Ammonium and Triethanolamine as Soaps—Determine (a) total alkalinity by the titration method, (b) total acidity, (c) free-and-alkali-combined fatty acids, and (d) total ammonia. The following results may be calculated:

Free fatty acids = (c) minus (a) = (f)
Ammonia plus triethanolamine as soap = (b) minus (f) = (g)
Ammonia as soap = (d)
Triethanolamine as soaps = (g) minus (d)
Fixed alkalinity as soap = (a) minus (g)

Sodium, Ammonium and Triethanolamine as Soaps, and Sodium Carbonate—Determine (a) total alkalinity by titration method, (b) free-and-alkali-combined fatty acids, (c) total ammonia, and (d) total alkalinity by the ash method. The following results may be calculated:

Alkalinity as sodium carbonate = (a) minus (b) = (f)
Ammonia as soap = (c)
Fixed alkalinity plus triethanolamine-alkalinity as soap = (b) minus (c) = (g)
Fixed alkalinity as soap = (d) minus (f) = (h)
Triethanolamine-alkalinity as soap = (g) minus (h)

Sodium Soap, Sodium Carbonate and Acetate—Determine (a) free-and-alkali-combined fatty acids, (b) alkalinity by the active ingredients methods, and (c) alkalinity either by the oleic acid method or by the ash method. These data give the following results:

Alkalinity as soda soap = (a)
Alkalinity as carbonate = (b) minus (a) = (d)
Alkalinity as acetate = (c) minus (b)

Sodium as Soap and Ammonium Sulfate or Chloride—Determine (a) total alkalinity by titration and (b) free-and-alkali-combined fatty acids, from which the following results may be calculated:

Fixed alkalinity as soap = (a)
Free fatty acids = (b) minus (a)

QUALITATIVE TEST FOR TRIETHANOLAMINE

Commercial triethanolamine gives a strong blue color with copper sulfate, identical with that produced by free ammonia. The following test used in the writer's laboratory, is based on that principle and was found to be sensitive to less than 0.5 per cent of triethanolamine based on the weight of the sample. The sample is mixed with concentrated salt solution and ether, and slightly acidified. The salt layer is then boiled with a solution of copper sulfate and strong caustic soda until all the ammonia, if any, is expelled. A distinct blue color of the filtered solution indicates triethanolamine. Ammonia does not interfere with the method.

Table I—Committee Results* of Alkalinities and Acidities on Sample E (Sulfonated Castor Oil Free of Ammonia)

	Total Alkalinity: Titration Method, Mg KOH/gm	*Total Acidity (Free Fatty Acids), Mg KOH/gm*	*Free and Alkali-Combined Fatty Acids, Mg KOH/gm*
Clark	31.86	33.56	64.20
Hart	31.76	33.79	65.35
Ford	31.70	33.66	65.36
Fowler	31.57	33.74	65.87
Gulick	31.22	33.33	....
Pingree	31.83	33.74	65.30
Silverman	31.85	33.85	65.25
Williams	32.16	33.62	65.34
Grand average	31.74	33.66	65.24
Calculated	....	....	65.40
Greatest difference.	0.94	0.52	1.67
Average deviation from mean	0.19	0.13	0.30

*Averages only are given.

COMMITTEE RESULTS

Two samples of sulfonated castor oil, namely, Sample E and Sample E-1, were analyzed by the committee for acidities and alkalinities. Sample E-1 was a mixture of two parts of Sample E (free of ammonia) and one part of ammonia water, the latter containing 1.56 per cent of NH_3 or 51.36 mgs as KOH per gram. The following determinations were made: (a) total alkalinity by the titration method, (b) free-and-alkali-combined fatty acids, (c) total acidity, and (d) in the case of Sample E-1, total ammonia. The procedures are given at the end of this report. The results for Sample E are given in Table I and for Sample E-1, in Table II. A discussion of the findings follow:

Total Alkalinity—The average deviation for both samples were approximately equal, namely, 0.19 to 0.20 mgs as KOH. The average alkalinity of Sample E-1, containing ammonia, was 38.36 mgs, compared with the calculated value 38.28 mgs, or a difference of 0.08 mgs, indicating that the method gives quantitative results for total alkalinity in the presence of ammonia soap.

Free-and-Alkali-Combined Fatty Acids—The average deviations for Samples E and E-1 were 0.30 mgs. and 0.39, respectively. The comparatively good agreement between the actual and calculated values will be noticed and incidentally offers additional proof that the total active ingredients may be extracted quantitatively and without decomposition.

Total Acidity—The average deviation for both samples were approximately the same, namely, 0.12 to 0.13 mgs. In the case of Sample E-1 the difference between the actual and calculated results was 0.77 mgs, which accuracy is considered satisfactory for commercial work and is proof that ammonia soap may be titrated as fatty acids with a fair degree of accuracy.

Table II—Committee Results* of Alkalinities and Acidities on Sample E-1 (Sulfonated Castor Oil Partly Neutralized with Ammonia)

	Total Alkalinity: Titration Method, Mg KOH/gm	*Free and Alkali-Combined Fatty Acids, Mg KOH/gm*	*Total Acidity, Mg KOH/gm*	*Alkalinity of Ammonia as Soap, Mg KOH/gm*	*%*	*Acidity as Free Fatty Acids, Mg KOH/gm*	*Fixed Alkalinity as Soap, Mg KOH/gm*	*Total Ammonia Mg KOH/gm*	*%*
Clark	38.09	....	23.38	....	...	...	....	16.22	0.49
Hart	38.53	43.76	23.20	17.97	0.54	5.23	20.56	16.90	0.51
Ford	38.24	43.61	23.39	18.02	0.55	5.37	20.22	17.49	0.53
Fowler	38.57	44.78	22.89	16.68	0.51	6.21	21.89	17.22	0.52
Gulick	37.96	44.37	23.17	16.76	0.51	6.41	21.20	16.95	0.51
Pingree	38.50	43.46	23.05	18.09	0.55	4.96	20.41	18.33	0.56
Silverman	38.40	44.00	23.20	17.60	0.53	5.60	20.80	19.20	0.58
Williams	38.61	44.44	23.20	17.37	0.53	5.83	21.24	16.59	0.50
Grand average	38.36	44.06	23.18	17.50	0.53	5.66	20.90	17.36	0.53
Calculated	38.28	43.60	22.47	17.12	0.52	5.32	21.16	17.12	0.52
Difference	0.04	0.46	0.71	0.38	0.01	0.34	0.26	0.24	0.01
Greatest difference	0.65	1.17	0.50	1.41	0.04	1.45	1.67	2.98	0.09
Average deviation from mean	0.20	0.39	0.12	0.53	0.02	0.45	0.47	0.73	0.02

*Averages only are given.

Ammonia —Excellent agreement was obtained for ammonia by the boiling method. The average deviation was only 0.02 per cent and the difference from the calculated value, only 0.01 per cent.

COMMENTS AND SUGGESTIONS

S. K. Ford: I believe this series of determinations is very useful in judging the value of a sulfated oil. The determinations of fixed alkali and free fatty acids are of importance, not only in the part played by these groups in affecting the properties of the oil, but also as a check upon the uniformity of various lots. The standard methods proposed by the committee, I believe to be excellent. I would also suggest that a qualitative test for triethanolamine and acetate be included in the report.

Arthur L. Fowler: It appears that ammonium sulfonate in alcoholic solution only partially titrates as fatty acids (approximately 25 per cent). The qualitative test for triethanolamine outlined in your letter of the 13th, has been used for sometime in this laboratory with success. It was originally, I believe, suggested by Joseph Zaparanick of L. Sonneborn & Sons, about five years ago and reported in the *Chemist-Analyst* (J. T. Baker & Co. publication). We have tried a series of amines using this test and it seems to be specific for triethanolamine (mono- and di- also give the test).

Gulick—Segessemann: We believe the methods given in the Committee Report are theoretically sound as well as practical. We wish to call attention to the fact that the qualitative test for ammonia, included in this report, will give positive results for mono- di- and triethanolamine.

Ralph Hart: The estimation of alkalinity and acidity in the usual grade of a sulfonated oil is a matter of two or three simple determinations. The proposed methods of the committee, however, are intended to include more complicated mixtures. Sodium acetate, found in many of the newer sulfonated oils which are usually highly sulfonated, interferes with the total alkalinity determination by the usual methods. Hence, the estimation of total alkalinity by the ash-method is a very useful addition to the procedures.

Mr. Pingree's suggestion to determine free fatty acids over brine solution is an excellent one, particularly useful in the case of dark colored oils, and has been incorporated in the methods.

That the alkalinity of triethanolamine soap may be determined quantitatively by titrating its water solution with methyl orange and the acidity of its soap, in alcoholic solution in presence of phenolphthalein are shown by the following experiment. A sample of sulfonated castor oil of known alkalinity and acidity was mixed with a known amount of triethanolamine dissolved in water, and the mixture analyzed. The results were as follows:

	Alkalinity as Triethanolamine Soap, mg. KOH/g		*Acidity as Triethanolamine Soap, mg. KOH/g*	
	Present	*Found*	*Present*	*Found*
Hart	13.5	13.7	13.5	13.9
Segessemann .	13.5	12.7	13.5	14.7
Silverman ...	13.5	13.0	13.5	13.2

R. A. Pingree: In the determination of free fatty acids, I find that it is not necessary to titrate in alcohol. After titrating for Total Alkalinity, the Free-and-Combined Fatty Acids may be determined on the same sample simply by adding 25 cc. of alcohol, to prevent jelling, and titrating back to phenolphthalein end point with 0.5 N NaOH. A very sharp end point is obtained by observing the lower brine layer. The Total Acidity may be determined directly in the same manner. Results check very well with the alcohol titration method. The brine or new method has two decided advantages over the alcohol titration method. First, considerable time is saved and second, the salt layer remains quite colorless, allowing a much sharper end point. This last advantage is particularly apparent with dark colored oils which frequently make it impossible to get a sharp end point when titrated in alcohol. This procedure, however, does not work satisfactorily in the present of ammonium compounds.

Excellent check results were obtained by this method as well as good agreement with the alcohol-titration method, as shown by the following results:

Total Acidity: Direct Methods, mg. KOH/g

	Brine Method	*Alcohol Method*
	33.79	33.81
	33.73	33.67
Average	33.76	33.74

Free-and-Combined Fatty Acids, mg. KOH/g

	65.49	65.13
	65.51	65.45
	65.54	....
Average	65.51	65.30

It seems that during the extraction of the acidified oil in ether with salt solution, all of the ammonia combined as sulfonate is split off from the oil. To demonstrate this point, I sulfonated a small amount of olive oil, washed it with ammonium sulfate and finally neutralized with ammonia. No sodium salt was in contact with the oil at any time. A sample of this oil was dissolved in ether and extracted five times with 25 cc. portions of a 25 per cent solution of NaCl. The resultant oil gave no test for NH_3 when boiled with excess caustic soda.

In the procedure for Total Alkalinity I suggest that in addition to 25 ml. of ether that also 25 ml. of alcohol be added. I find that this addition of alcohol prevents jelling and hastens the titration considerably by causing the layers to separate faster. In this way the end point may be readily obtained without the necessity of titrating back.

I would suggest that "Combined Fatty Acids" be replaced by "Fatty Acids Combined as Soap." I also

think that it would be well to omit the word "titer" from the calculations. I do not believe that it is commonly used in this respect now. Why not let it read "ml. 0.5N × 28.055" or whatever the case may be? Under Total Alkalinity, 4 or 5 drops of phenolphthalein solution is ample.

I believe that the biuret reaction with triethanolamine will be a suitable test. Glycerine also gives this same reaction. However, I find that it does not interfere if present in the amounts which are usually encountered in sulfonated oils.

WILLIAMS—JAREK: We can offer no serious criticism on the method proposed for the determination of sodium soap and ammonium soap in sulfonated oils. The procedure appears theoretically sound; the only objectionable feature being the long and drawn out method necessary to arrive at the results desired. On the other determinations and calculations of acidities and alkalinities mentioned in your letter, there is no criticism to be offered.

SILVERMAN—MAZIER: In general, we wish to state that in our laboratory we have worked quite a good deal with triethanolamine alkalinity and acidity. We have always found that triethanolamine soaps can be quantitatively titrated in water solution and that triethanolamine soap in alcohol can be quantitatively titrated as free fatty acids in the presence of phenolphthalein.

SUMMARY AND RECOMMENDATIONS

In this report various methods for the determination of alkalinities and acidities in sulfonated oils have been discussed, and methods of analysis for the more complicated mixtures outlined. The committee has investigated experimentally the more promising procedures and, as a result of this work, the committee proposes as standard methods the following procedures (given in detail at the end of this report):

I. QUALITATIVE TEST FOR AMMONIA
II. QUALITATIVE TEST FOR TRIETHANOLAMINE
III. ALKALINITIES
  A. Total Alkalinity: Titration Method
  B. Total Alkalinity: Active Ingredients Method
  C. Total Alkalinity: Ash-Method
  D. Total Alkalinity: Oleic-Acid Method
  E. Alkalinity as Ammonia or Triethanolamine: Free and/or as Soap
  F. Fixed Alkalinity as Soap
IV. ACIDITIES AND FREE ALKALI
  A. Total Acidity
  B. Acidity as Free Fatty Acids
  C. Acidity as Free Fatty Acids: Brine Method
  D. Acidity as Free Fatty Acids: Indirect Method
  E. Free Caustic Alkali
V. FREE-AND-ALKALI-COMBINED FATTY ACIDS
VI. TOTAL AMMONIA

I—PROPOSED QUALITATIVE TEST FOR AMMONIA IN SULFONATED (SULFATED) OILS

Equal parts of the sample, glycerol or diethylene glycol, and 25 per cent solution of caustic soda shall be mixed, transferred to a test tube, and heated in a hot-water bath. The escaping vapors shall be carefully tested for ammonia with moistened red litmus paper.

II—PROPOSED QUALITATIVE TEST FOR TRIETHANOLAMINE IN SULFONATED (Sulfated) OILS

About 10 grams of the sample shall be mixed in a separatory funnel with 50 ml. of a saturated solution of sodium chloride and 50 ml. of ether, and 5 drops of 0.1 per cent solution of methyl orange and N/2 sulfuric acid added until slightly in excess. The mixture shall be shaken thoroughly, the layers allowed to separate, and the water layer extracted again with 30 ml. of ether. The water layer shall now be heated to expel ether; to 25 ml. of the ether-free solution, there shall be added 1 ml. of a 5 per cent solution of copper sulfate crystals, 3 ml. of a 25 per cent solution of sodium hydroxide, and the solution boiled for 5 to 10 minutes. If ammonia is also present, the solution shall be boiled—if necessary with further addition of alkali—until all the ammonia has been expelled (test vapors with moistured red litmus paper). A distinct blue color after the boiling indicates the presence of triethanolamine. If a black, brown or green precipitate forms, the solution shall be filtered and the filtrate examined for color. A blank with 25 ml. of saturated sodium chloride solution shall be run simultaneously.

III—PROPOSED STANDARD METHODS OF TEST FOR ALKALINITIES IN SULFONATED (SULFATED) OILS

A. TOTAL ALKALINITY: TITRATION METHOD

Scope:

1. This method of test determines the total alkalinity existing in a sample of sulfonated (sulfated) oil by titrating a water-solution of the sample with mineral acid in the presence of methyl orange as the indicator. This method determines the alkalinity of the fixed alkali, ammonia, and triethanolamine bound as soap, and the alkalinity of titratable alkaline salt but not that of non-titratable alkaline salts.

Procedure

General:

2. The procedure shall consist of titrating a water-solution of the sample with mineral acid until neutral to methyl orange.

3. Ten grams of the sample shall be dissolved in 100 ml. of water contained in a 250-ml. glass-stoppered Erlenmeyer flask, warming to obtain solution if necessary. After cooling, 30 grams of sodium chloride, 25 ml. of ether, 25 ml. of neutral alcohol, and 5 drops of methyl orange indicator (0.1 per cent water-solution) shall be added, and

enough of half-normal sulfuric or hydrochloric acid added, with frequent but gentle shaking, until the mixture is slightly on the acid side. The contents shall now be shaken vigorously, and the titration completed by adding first half-normal sodium or potassium hydroxide—several drops at a time—until the solution is alkaline and then the acid, one or two drops at a time, until the end point is reached, shaking vigorously after each addition of reagent.

NOTE: If there is a tendency to jell during the titration, 25 ml. of neutral alcohol shall be added.

Calculation

Total Alkalinity—Titration Method:

4. The number of ml. of the acid multiplied by its equivalent as mgm. KOH minus the number of ml. of the alkali times its equivalent as mgm. KOH and divided by the weight of the sample shall be the total alkalinity of the sample, in mgm. KOH per gram of sample, or

$$\text{Total alkalinity, mgm. KOH/gm} = \frac{(\text{ml. acid} \times \text{equivalent as mgm. KOH}) \text{ minus } (\text{ml. alkali} \times \text{equivalent as mgm. KOH})}{\text{weight of sample}}$$

and shall be reported as ". . . mgm. KOH per gram of sample, A.A.T.C.C. Titration Method."

B. TOTAL ALKALINITY: ACTIVE INGREDIENT METHOD

Scope:

1. This method of test determines the total alkalinity existing in a sample of sulfonated (sulfated) oil by acidifying a water-solution of the sample with a known quantity of mineral acid, extracting the active ingredients, and completing the titration of the water layer. This method determines the alkalinity of the fixed alkali, ammonia, and triethanolamine bound as soap, and the alkalinity of titratable salts. Ammonium sulfate or chloride, or sodium acetate do not interfere with this method.

Procedure

General:

2. The procedure shall consist of acidifying a water-solution of the sample with a known quantity of sulfuric acid, extracting the active ingredients, and completing the titration of the water layers, using phenolphthalein as the indicator.

Water-Layer and Combined Wash Waters:

3. The active ingredients shall be extracted by the A.A.T.C.C. Total Active Ingredients Method[7], except that the quantity of acid required to acidify the sample shall be noted and the water-layers and the wash-waters shall be combined and saved.

Titration of Water-Layers:

4. The combined water-layers shall be gently heated under a reflux condenser to expel carbon dioxide, and then titrated until neutral to phenolphthalein.

Calculation

Total Alkalinity: Active Ingredients Method:

5. The number of ml. of the acid times its equivalent in mgm. KOH, minus the number of ml. of the alkali times its equivalent in mgm. KOH, and divided by weight of the sample shall be the total alkalinity of the sample in mgm. KOH per gram of sample, or

$$\text{Total alkalinity, mgm. KOH/g} = \frac{(\text{ml. acid} \times \text{equivalent as mg. KOH}) \text{ minus } (\text{ml. alkali} \times \text{equivalent as mg. KOH})}{\text{weight of sample}}$$

and shall be reported as ". . . mgm. KOH per gram of sample, A.A.T.C.C. Active Ingredients Method.

C. TOTAL ALKALINITY: ASH METHOD

Scope:

1. This method of test determines the alkalinity existing in a sample of sulfonated (sulfated) oil by ashing the sample, with or without the addition of alkali, and determining the alkalinity of the ash. This method determines the combined alkalinities of fixed alkali-soaps and both of titratable and non-titratable alkaline salts, such as sodium carbonates, sodium acetate, etc. Ammonia and/or triethanolamine soaps and ammonium sulfate or chloride do not interfere with this method.

Procedure

General:

2. The procedure shall consist of three determinations, namely, (a) the organically combined SO_3 by the ash-gravimetric method, (b) the alkalinity of the sample after ignition, and (c) ammonia as ammonium sulfate or chloride.

Organically Combined SO_3:

3. The organically combined SO_3 shall be determined according to the A.A.T.C.C. ash-gravimetric method[5].

Alkalinity of Ash:

4. Ten grams of the sample shall be mixed in a crucible, provided with a small glass stirring rod, with about 5 ml. of neutral alcohol and with a slight excess of normal sodium hydroxide sufficient: (a) to saponify the free fatty acids, fatty acids combined as ammonium soap, and neutral fat, and (b) to replace all the ammonia, if any, combined as ammonium sulfate or chloride. The mixture shall be gently heated for about 15 minutes, then evaporated to dryness, the stirring rod wiped clean with ashless filter paper which shall be added to the crucible, the contents gently burned, and finally ignited until the carbon is nearly consumed. The residue shall be treated with a few drops of 30 per cent neutral hydrogen peroxide and ignited again. The treatment with the peroxide shall be repeated until the residue is practically free from carbon.

The residue shall be dissolved in hot water, 5 drops of methyl orange solution added, and treated with half-normal acid until a slight excess is present. The solution shall now be gently boiled, to expel carbon dioxide, and the titration completed in the usual manner.

Ammonia as Ammonium Sulfate or Chloride:

5. The ammonia as ammonium sulfate or chloride shall

be determined as under "Ammonia as Ammonium Sulfate or Chloride" (given below).

Calculation

Total Alkalinity—Ash Method (In Absence of Ammonium Sulfate, Chloride, etc.):

6. The total alkalinity of the sample, in mgms. KOH per gram of sample, shall be calculated according to the following formula:

$$\text{Total Alkalinity, mgm. KOH/g} = \frac{(\text{ml. acid} \times \text{equiv. as mgm. KOH}) \text{ minus (alkali added})}{\text{weight of sample}}$$

plus (7.013 × per cent Combined SO_3)

and shall be reported as ". . . mgm. KOH per gram of sample, A.A.T.C.C. Ash Method."

Total Alkalinity—Ash Method (In the presence of Ammonium Sulfate, Chloride, etc.):

7. The total alkalinity of the sample, in mgms. KOH per gram of sample shall be calculated according to the following formula:

$$\text{Total Alkalinity, mgm. KOH} = \frac{(\text{ml. acid} \times \text{equiv. as mgm. KOH}) \text{ minus (alkali added})}{\text{weight of sample}}$$

plus (7.013 × Combined SO_3) plus (33.0 × per cent NH_3 as ammonium salts)

and shall be reported as ". . . mgm. KOH per gram of sample, A.A.T.C.C. Ash-Method."

D. TOTAL ALKALINITY: OLEIC ACID METHOD

Scope:

1. This method of test determines the alkalinity existing in a sample of sulfonated (sulfated) oil by heating the sample with excess oleic acid, acidifying a water-solution of the heated sample with a known quantity of mineral acid, extracting the active ingredients, and completing the titration of the water-layer. This method determines the alkalinity of the fixed alkali, ammonia, and triethanolamine bound as soap, and of titratable salts and sodium acetate.

General:

2. The procedure shall consist of heating the sample with oleic acid until practically free of acetic acid, acidifying with a known quantity of sulfuric acid, extracting the active ingredients, and completing the titration of the water-layer, with methyl orange as the indicator.

Expelling Acetic Acid:

3. Five grams of the sample shall be mixed with double its weight of oleic acid in a 250 ml. beaker and heated in an oil-bath with constant stirring for 30 minutes at 123° to 125° C.

Extracting the Active Ingredients:

4. The cooled sample shall be mixed with 50 ml. of saturated salt solution and 50 ml. of ether, and acidified with a known amount of 0.5 N sulfuric acid until the mixture is distinctly acid to methyl orange (about 0.2 ml. in excess). The water-layer shall be repeatedly extracted with 40 ml. portions of ether until the latter reacts neutral to phenophthalein.

Titration of Water-Layers:

5. The titration of the combined water-layers shall then be completed, using methyl orange as the indicator.

Calculation

Total Alkalinity: Oleic Acid Method:

6. The same as Active Ingredient Method.

E. ALKALINITY AS AMMONIA AND/OR TRIETHANOLAMINE-FREE AND/OR AS SOAP

Scope:

1. This method of test determines the alkalinity of ammonia and/or triethanolamine free or bound as soap, existing in a sample of sulfonated (sulfated) oil by calculation from the total alkalinity, acidity, and free-and-combined fatty acids. The method is not applicable in the presence of ammonium sulfate or other acid salts.

Procedure

General:

2. The procedure shall consist of three determinations, namely, (a) total alkalinity, (b) total acidity, and (c) free-and-alkali combined fatty acids.

Total Alkalinity:

3. Total alkalinity shall be determined as under "Total Alkalinity: Titration Method."

Total Acidity:

4. Total acidity shall be determined as under "Total Acidity."

Free-and-Alkali-Combined Fatty Acids:

5. Free-and-alkali-combined fatty acids shall be determined as under "Free-and-alkali-combined Fatty Acids."

Calculation

Alkalinity as Ammonia and/or Triethanolamine, Free and as Soap:

6. The alkalinity of ammonia or triethanolamine, free and as soap, in mgm. KOH per gram of sample, shall be the sum of the total alkalinity and total acidity minus the free-and-combined fatty acids, or

> Alkalinity as ammonia and/or triethanolamine, free and combined as soap, mgm. KOH/g = total alkalinity plus total acidity minus free-and-combined fatty acids

and shall be reported as ". . . mgm. KOH/g, A.A.T.C.C. Calculation Method."

Alkalinity as Free Ammonia and/or Triethanolamine:

7. The alkalinity of free ammonia and/or triethanolamine, in mgm. KOH per gram of sample, shall be the total alkalinity minus the free-and-alkali-combined fatty acids, or

> Alkalinity of free ammonia and/or triethanolamine, mgm. KOH/g = total alkalinity minus free-and-alkali-combined fatty acids

and shall be reported as ". . . mgm. KOH/g A.A.T.C.C. Calculation Method."

Alkalinity of Ammonia and/or Triethanolamine as Soap:

8. The alkalinity of ammonia and/or triethanolamine as

soap in mgm. KOH/g per gram of sample, shall be the difference between the "alkalinity as ammonia and/or triethanolamine, free and as soap," and the "alkalinity as free ammonia or triethanolamine," or

Alkalinity of ammonia or triethanolamine as soap, mgm. KOH/g = alkalinity "free and as soap" minus alkalinity "free"

and shall be reported as ". . . mgm. KOH/g A.A.T.C.C. Calculation Method."

F. FIXED ALKALINITY AS SOAP: INDIRECT METHOD

Scope:

1. This method of test determines the fixed alkalinity bound as soap existing in a sample of sulfonated (sulfated) oil by calculation from the total acidity, and free-and-alkali-combined fatty acids. It is applicable in the presence of ammonia or triethanolamine soaps but not acid salts such as ammonium sulfate.

General:

Procedure

2. The procedure shall consist of two determinations, namely, (a) total acidity, and (b) free-and-alkali-combined fatty acids.

Total Acidity:

3. Total acidity shall be determined as under "Total Acidity."

Free-and-Alkali-Combined Fatty Acids:

4. Free-and-alkali-combined fatty acids shall be determined as under "Free-and-Alkali-Combined Fatty Acids."

Calculation

Fixed Alkalinity as Soap:

5. The fixed alkalinity as soap, in mgm. KOH per gram of sample, shall be the free-and-alkali-combined fatty acids minus the total acidity, or

Fixed alkalinity as soap, mgm. KOH/g = free-and-alkali-combined fatty acids minus total acidity

and shall be reported as ". . . mgm. KOH/g A.A.T.C.C. Method."

IV—PROPOSED STANDARD METHODS OF TEST FOR ACIDITIES AND FREE ALKALI IN SULFONATED (Sulfated) OILS

A. TOTAL ACIDITY

Scope:

1. This method of test determines the acidity existing in a sample of sulfonated (sulfated) oil by titrating the sample dissolved in a solvent in the presence of phenolphthalein indicator. This method is not applicable in the presence of acid-reacting salts, such as ammonium sulfate, ammonium sulfonate, etc.

General:

Procedure

2. The procedure shall consist of titrating a solution of the sample in a mixture of alcohol and ether in the presence of phenolphthalein indicator.

Total Acidity:

3. Ten grams of the sample shall be dissolved in a mixture of 50 ml. of neutral alcohol and 25 ml. of ether, 5 drops of phenolphthalein indicator (1.0 per cent alcohol solution) added, and the contents titrated with half-normal sodium or potassium hydroxide until the pink color persists after vigorous shaking.

Calculation

Total Acidity:

4. The total acidity, in mgm. KOH per gram of sample, shall be equal to the number of ml. of the alkali times its equivalent as mgm. KOH, divided by the weight of the sample, or

Total acidity, mgm. KOH/g = ml. alkali × equivalent as mg. KOH/wt. of sample.

Method."

B. ACIDITY AS FREE FATTY ACIDS

Scope:

1. This method of test determines the acidity as free fatty acids existing in a sample of sulfonated (sulfated) oil by titrating the sample dissolved in a mixture of alcohol and ether until neutral to phenolphthalein. It is not applicable in the presence of ammonium or triethanolamine soaps or acid salts.

Procedure

Procedure:

2. The procedure shall be the same as under "Total Acidity."

Calculation

Acidity as Free Fatty Acids:

3. The acidity as free fatty acids shall be calculated and reported as under "Total Acidity."

C. ACIDITY AS FREE FATTY ACIDS: ALTERNATE (BRINE) METHOD

Scope:

1. This method of test determines the acidity as free fatty acids existing in a sample of sulfonated (sulfated) oil by titrating a water-solution of the sample with phenolphthalein as the indicator. It is not applicable in the presence of ammonia or triethanolamine soaps or acid salts.

Procedure

General:

2. The procedure shall consist of titrating a water-solution of the sample in the presence of salt, ether and alcohol, with phenolphthalein as the indicator.

Acidity as Free Fatty Acids:

3. Ten grams of the sample shall be dissolved in 100 ml. of water contained in a 250-ml. glass-stoppered Erlenmeyer flask, warming to obtain solution if necessary. After cooling, 30 grams of sodium chloride, 25 ml. of ether, 50 ml. of neutral alcohol, and 5 drops of phenolphthalein indicator (1.0 per cent alcohol solution) shall be added.

NOTE: At this stage methyl orange indicator may be added and total alkalinity determined as under "Total Alkalinity: Titration Method."

The contents shall now be titrated with 0.5 N sodium

hydroxide until the first pink tinge appears in the water layer, shaking vigorously after each addition of the reagent.

Calculation

Acidity as Free Fatty Acids:

4. The acidity as free fatty acids shall be calculated and reported as under "Total Acidity."

NOTE: In case the total alkalinity was determined on the same sample, the free fatty acids shall be calculated as follows:
Acidity as free fatty acids, mg. KOH/g= ml. alkali × equivalent as mg. KOH/wt. of sample minus (total alkalinity).

D. ACIDITY AS FREE FATTY ACIDS: INDIRECT METHOD

Scope:

1. This method of test determines the acidity as free fatty acids in a sample of sulfonated (sulfated) oil by calculation from total alkalinity and free-and-alkali-combined fatty acids. It is applicable in the presence of ammonia or triethanolamine soaps but not of their salts or other acid salts.

Procedure

General:

2. The procedure shall consist of two determinations, namely, (1) total alkalinity and (2) free-and-alkali combined fatty acids.

Total Alkalinity:

3. Total alkalinity shall be determined as under "Total Alkalinity: Titration Method."

Free-and-Alkali-Combined Fatty Acids:

4. Free-and-alkali-combined fatty acids shall be determined as under "Free-and-Alkali-Combined Fatty Acids."

Calculation

Acidity as Free Fatty Acids:

5. Acidity as free fatty acids, in mgm. KOH per gram of sample, shall be the free-and-alkali combined fatty acids minus the total alkalinity, or

Acidity as free fatty acids, mgm. KOH/g= Free-and-alkali-combined fatty acids minus total alkalinity

and shall be reported as ". . . mgm. KOH/g A.A.T.C.C. Calculation Method."

E. FREE CAUSTIC ALKALI

Scope:

1. This method of test determines the free caustic alkali in a sample of sulfonated (sulfated) oil by titrating the sample dissolved in a mixture of alcohol and ether until neutral to phenolphthalein.

Procedure

Procedure:

2. The procedure shall be the same as under "Total Acidity," with the exception that half-normal acid shall be used in the titration.

Calculation

Free Alkali:

3. The free caustic alkali, in mgm. KOH per gram of the sample, shall be the number of ml. of acid times its equivalent as mg. KOH titer divided by the weight of the sample, or

Free caustic alkali, mgm. KOH/g = ml. acid × equivalent as mg. KOH/weight of sample.

and shall be reported as ". . . mgm. KOH/g."

V—PROPOSED STANDARD METHOD OF TEST FOR ACIDITY AS FREE-AND-ALKALI-COMBINED FATTY ACIDS

Scope:

1. This method of test determines the free-and-alkali-combined fatty acids in a sample of sulfonated (sulfated) oil by extracting the active ingredients and determining the total free fatty acids in the extract by titration.

Procedure

General:

2. The procedure shall consist of extracting the active ingredients and determining the total acidity of the extract.

Active Ingredients:

3. The active ingredients shall be extracted by the A.A.T.C.C. Total Active Ingredients Method[7], except that dehydration of the extract shall be omitted:

NOTE: In the presence of acetate, the ether layer shall be carefully washed with brine solution until one or two drops of N/2 of caustic alkali to the final wash water is neutral to phenolphthalein indicator.

Total Acidity:

4. Exactly 0.5 ml. of half-normal caustic alkali shall be added to the extract containing the total active ingredients, the ether evaporated to a volume of about 25 ml., about 50 ml. of neutral alcohol added, and the mixture titrated as under "Total Acidity."

In the case of dark colored oils that may interfere with the end point, the mixture after the bulk of the ether has been evaporated shall be titrated as under "Acidity as Free Fatty Acids: Brine Method," after the addition of water, salt, and alcohol.

Calculation

Free-and-Alkali-Combined Fatty Acids:

5. The free-and-alkali-combined fatty acids shall be calculated and reported as under "Total Acidity."

VI—PROPOSED STANDARD METHOD OF TEST FOR TOTAL AMMONIA IN SULFONATED (Sulfated) Oil

TOTAL AMMONIA

Scope:

1. This method of test determines the total ammonia in a sample of sulfonated (sulfated) oil by boiling a water-solution of the sample with excess alkali and determining by titration the loss in alkali after the boiling.

Procedure

General:

2. The procedure shall consist of two determinations, namely, (1) the total alkalinity and (2) the loss in alkalin-

ity after boiling with excess sodium hydroxide.

Total Alkalinity:

3. The total alkalinity shall be determined as under "Total Alkalinity: Titration Method."

Alkalinity After Boiling:

4. Ten grams of the sample shall be dissolved in 100 ml. of water in a 500 ml. beaker, 25 ml. of half-normal sodium hydroxide added, and the mixture boiled for half an hour, or until all the ammonia is expelled (moistened red litmus paper). The contents shall be cooled, methyl orange indicator (0.1 per cent solution) added, and titrated to an approximate end point. The mixture shall now be transferred to a 250-ml. glass-stoppered flask, and the titration completed (with the addition of salt and ether) as under "Total Alkalinity: Titration Method."

Calculation

Total Ammonia:

5. The total ammonia in the sample, in mgm. KOH per gram, shall be the total alkalinity plus the difference between the number of ml. of alkali added times its equivalent as mg. KOH and the number of ml. of acid times its equivalent as mg. KOH, this difference first to be divided by the weight of the sample, or

Total ammonia, mgm. KOH/g=
total alkalinity plus
(ml. alkali added × equivalent as mg. KOH minus ml. acid × equivalent as mg. KOH/wt. of sample)

and shall be reported as ". . . mgm. KOH, A.A.T.C.C. Method."

6. To convert ammonia from its equivalent of mgm. KOH per gram of sample into per cent ammonia, the following formula shall be used:

Per cent NH_3 = 0.0303 times
(ammonia as mgm. KOH per gram).

LITERATURE CITED

[1]Commercial Standard CS43-32, Bureau of Standards, Washington, D. C. (1933).
[2]Fredchenka and Kashin, Masloboino Zhirovoe Delo 11, 498 (1935); also Chem. Abst., 30, 892 (1936).
[3]Hart R., Ind. Eng. Chem., 9, 850 (1917).
[4]Ibid, Anal. Ed., 4, 411 (1932).
[5]Ibid 22, 695 (1933).
[6]Hart, R., et al, Am. Dyestuff Reptr., 22, Nov. 20 (1933).
[7]Ibid. 23, May 21 (1934).
[8]Ibid, 24, May 20 (1935).
[9]Kertesz, Z. I., Magyar Chem. Folyoirat, 33, 135 (1927).
[10]"Methods of the A.L.C.A.," Am. Leather Chem. Soc., Ridgeway, Pa., May (1930).
[11]"Methods of Analysis," Assoc. Off. Agr. Chem., Third Ed., 326 (1930).
[12]Sheely, M. L., et al, Oil and Soap, 11, 90, May (1934).
[13]Sors, Paul, Chem.-Ztg., 56, 156 (1932).
[14]Untersuchung von Türkischrotölen and türkischrotölartigen Produkten, Wissenschaftliche Verlagagesellschaft m.b.H., Brochure (1930); also Am. Leather Chem. Assoc., 27, 52 (1932).

Problems in the Dyeing and Finishing of

Fabrics Containing Bemberg Yarn*

E. H. BENZING†

I BELIEVE all dyers have had occasion to dye Bemberg yarn in one form or another in their dyeing plants; either in skeins for the tie silk trade, or as a filling in crepe cloths with acetate warps, or in their printing plants they have had an occasion to print some of the Bemberg sheer cloth, and I know that you will be interested in the history and manufacture of the yarn. Bemberg yarn is made by the cuprammonium process invented in 1890 by Despeisses and was developed in England and Germany. In 1905 the J. P. Bemberg A. G. succeeded in working out the cuprammonium stretch spinning process and named the yarn so obtained "Bemberg".

In October 1926 the first spinning machine of the American Bemberg Corporation was started at Elizabethton, Tenn., which means also that on that day the first thread by the cuprammonium process was spun in the United States. The production and improvement of this product made such progress that after a few years the original plant was enlarged. Today there are Bemberg plants all over the world. Bemberg is now produced in the United States, Germany, France, England, Italy, and Japan. I have with me and will be glad to show you demonstration material illustrating the manufacture of Bemberg from raw material to finished yarn.

Like most synthetic fibers we use cellulose as a raw material, the cellulose that is found in cotton linters, the short silky fibers adhering to the cotton seed after the bulk of the cotton has been removed in the ginning process. No subsequent bleaching of Bemberg yarn is necessary, as the bleached cotton linters that are used remain white in the process of manufacture after the copper has been removed. Cellulose is treated more mildly in the cuprammonium process. The purified cotton linters are added to a solution of copper in concentrated ammonia, that is: finely divided copper is dissolved in concentrated ammonia until the necessary concentration is obtained, and

*Presented at meeting, New York Section, October 23, 1936.
†American Bemberg Corp.

to this is added a measured quantity of pure cotton. The cuprammonium spinning solution thus prepared is carefully filtered and stored until required for the spinning process.

CUPRAMMONUM SPINNING

After passing a succession of filters the spinning solution is forced through individual spinnerettes with rather large holes into a glass funnel containing a setting bath. On leaving the funnel the filaments pass through a bath of mild sulfuric acid and are then wound onto reels in the form of skeins. The concentration of the setting bath is purposely made so as to precipitate the cellulose slowly rather than almost instantaneously as in the viscose process. The slow coagulation allows the solution to be stretched tremendously during spinning, and it is from this step in the manufacture of cuprammonium yarn that the term "stretch spinning" is derived.

DECOPPERING AND FINISHING

Immediately after the spinning operation an acid rinse frees the yarn from all but minute traces of the copper. Following the acid rinse, the yarn is rinsed thoroughly, neutralized, and finished with soap and oil, extracted, and dried. The dried cuprammonium process yarn is then wound into the form of put-up desired by the trade. Unlike other synthetic yarns it contains no twist from the spinning and is sold in appreciable quantities in an untwisted condition.

UNEVENNESS IN FABRICS CONTAINING BEMBERG YARNS

They may originate with the designer who has made an error in the construction. They may be traced very often to a throwster who has given insufficient attention to the factors of moisture and tension. They may be due to the weaver who has neglected these same factors in copping the yarn for weaving, or who occasionally may load the warp with size which is too slowly removed and so absorbs the creping power of the filling in the boil-off vats before it can draw the softening warp ends into the desired pebble. Finally they may be due to the handling of the fabrics in a finishing plant, caused by stick marks, too sudden shrinkage, or insufficient removal of size (due to its fine filaments Bemberg requires more attention in the removal of size).

DYE DEFECTS

Uneven dyeing may appear as irregular stains or blotches, as filling bars or as warp streaks. There are dye streaks due to variations in the yarn. They are rarer now than a few years ago because of the careful control which the yarn manufacturers have evolved and applied to every step of their processes. There are streaks which are due to faulty selection of dyestuffs, or to faulty treatment in boil-off or in the dye bath. There are blotches caused by sizes and by throwing oils having ingredients that "break" out of their emulsions and fasten themselves in resist spots upon a fabric.

If some fabrics can be made right, all can be made right by rigid control of the conditions at each stage. The yarn producer had to adopt and to devise scientific methods of careful control. This same degree of exactness in manufacture must be applied by the throwster, the knitter, the weaver, and the finisher in order to meet the standard of the trade.

It is a known fact that fabrics woven in some mills are relative to warp streaks far superior to those woven in other mills, which is more or less due to careful handling throughout the operations. Special attention must be given to controlled humidity and tension. When lining cloth is scoured in open form, dried loose, dyed on the pad (at high speed), definitely avoiding dry cans, and dried on the frame, it is possible to produce this cloth practically free from warp streaks.

By observing the proper procedure and using proper dyestuffs, etc., dyers can go a long way towards overcoming defects caused by uneven tension applied during fabric manufacturing operations. Crepe fabrics even more than flat fabrics show defects due to uneven tension and uneven humidity control. It is the general consensus of opinion that the dyer, for the most part, simply blames the throwster for cracks, pebble variations, etc., without attempting to go further into the problem. The fact of the case is that, if dyers had not of necessity designed special processes for fabrics inherently faulty due to uneven tension during throwing (I refer to the so-called embossing process) hundreds of thousands, if not millions of yards, would definitely have been defective during the past two years. I have seen dyers making a salable piece of goods by overcoming troubles caused in fabric manufacture.

Whereas it is today generally believed by many people that the soaking bath variations have less effect on pebble than humidity, tension, etc., during throwing, those of us who have studied the subject carefully find that the soaking bath is an exceedingly important part of the production of proper crepe. Each of the various viscose yarns require special soakings to produce the best results and Bemberg yarn should be soaked quite differently from viscose in order to produce the pebble effect that is expected by the designer. Also a close cooperation with the dyer throughout the operations from raw yarn to finished fabric will result in a quick solution of the problem. Many thousands of yards of Bemberg-filled Canton crepe have been finished satisfactorily. There are no difficulties in connection with the printing of fabrics made from Bemberg yarn. It must only be remembered that the goods should not be dried at a very high temperature after printing and that they should be washed immediately after steaming. I wish to say at this point that, if heated in a dry atmosphere for some time, regenerated cellulose yarn (viscose, cuprammonium as well as acetate yarns) gradually turn yellow and serious changes in the physical properties take place.

Bemberg is being successfully used in knitted cloths quite extensively for underwear, hosiery, gloves, tie silks and outerwear.

The major handicap is lack of knowledge of Bemberg on the part of designers, throwsters, weavers, dyers, and finishers. In the absence of definite information to the contrary, technicians often handle Bemberg like viscose, blaming the yarn rather than themselves when trouble is encountered. This can be remedied, if possible, by obtaining the necessary information from the yarn manufacturer who will assist in obtaining satisfactory results.

DISCUSSION

Chairman Baxter: I know Mr. Benzing will be very willing to answer questions. I feel sure, looking around this room, that we should have a lot of interesting questions from some of those present. As Mr. Benzing said, Bemberg is different from any of the other fibers; it has to go through all the mill, and all its peculiarities must be known before you handle that or any other rayon fabric.

In connection with boil-offs, Mr. Benzing, you said that your boil-off was different from that of any of the other types of rayon. Perhaps you might tell in what way it differs. I don't know whether everyone knows it or not.

Mr Benzing: What I meant is that it requires a little longer time due to more filaments. Naturally, the more filaments, the more size, for there are more filaments to get it out from, and in that way it just requires a longer time.

Mr. Lipol: Is ammonia recommended especially for scouring rayon—the presence of ammonia?

Mr. Benzing: It helps a good deal in the scouring, yes. In knitted fabrics particularly they use a lot of ammonia and soaps.

Mr. Lipol: It isn't necessary to use ammonia though, is it?

Mr. Benzing: No, not actually.

Mr. Smithberg: Are different sizing materials used for Bemberg?

Mr. Benzing: No, those that are used for viscose type yarns are used for the Bemberg.

Chairman Baxter: Isn't some of the trouble that the finishers experience due to the fact that they don't know what sizing compounds have been used by the mill?

Mr. Benzing: You really have to know what sizing compound is used. But even granted they are using the same size as they would on the viscose type yarn, it would require a little more time on Bemberg than on viscose.

Chairman Baxter: Are there any other questions?

Mr. Meili: Do you use oils or soap for scouring? Which do you prefer?

Mr. Benzing: We have had good results from both. I believe that a good soap boil-off has given satisfaction, and also oil boil-offs have given satisfaction. It all depends upon the individual.

Mr. Meili: Do you recommend a combination of the two?

Mr. Benzing: A combination is even better.

Mr. Meili: Do you recommend any enzymes?

Mr. Benzing: Not particularly, unless there is starch used in the sizing.

Mr. Meili: Gelatin is used a great deal at the present time and many dyers have difficulty in getting a complete removal of the sizing. Which do you recommend to use?

Mr. Benzing: I think it would assist in the quicker removal of the sizing. Naturally, the enzymes would have an influence on desizing the gelatin.

Chairman Baxter: Mr. Benzing spoke about some of the varied faults which occur in the dyeing of Bemberg. I am sure there are some dyers here that might have questions they would care to ask.

Mr. Miller: Is the caustic treatment necessary to swell the yarn?

Mr. Benzing: Are you speaking of sheer cloth?

Mr. Miller: No.

Mr. Benzing: No, it isn't necessary.

Mr. Dougan: Is it a fact that Bemberg dyes more quickly than viscose? Is there any relationship with regard to the absorption?

Mr. Benzing: You mean the percentage?

Mr. Dougan: Yes.

Mr. Benzing: Well, we all know dyers have had experience in dyeing viscose and cuprammonium yarn. With Bemberg the dye is absorbed much more quickly.

Mr. Dougan: Is there a definite relationship? For instance, take scarlet formyl: is it always a case of, say, one per cent? Suppose we have one fabric of viscose and one of Bemberg. I have noticed that the viscose is almost white and the Bemberg would be deep pink. Is there any definite relationship?

Mr. Benzing: Well, it all depends upon the chemist. I mean, as you say, I have seen cases where the Bemberg would be a dark pink and the viscose would be white—no dye at all. But if you raise the temperature you can get them almost alike.

Mr. Dougan: Is it possible to get it uniform?

Mr. Benzing: Yes, by boiling—constant boiling.

Chairman Baxter: A combination of the two is being dyed today in novelty fashions, and they are being dyed uniformly in the darker shades even.

Mr. Dougan: That has always been more or less of a problem.

Mr. Benzing: The temperature has a lot to do with it.

Mr. Barber: How much better is Bemberg today than it was two or three years ago, especially a crepe fabric with an acetate warp—a Bemberg crepe filler? Two or three years ago we couldn't get a regular creping on a crepe fabric from Bemberg yarn. Has that been overcome?

Mr. Benzing: With regard to creping of Bemberg, as I said before, I think the way the yarn is sized plays a very important part. We know that whereas with viscose

more twisted yarn is thrown with very little gelatin, with Bemberg we need a large percentage of gelatin, and we are even soaking yarn preparatory to throwing and in that way have had very satisfactory results in producing the desired pebble effect, by soaking it in different ways.

Mr. Barber: Then I am to understand from what you said that the Bemberg people recently, within the last two or three years, have begun to use more gelatin for the holding of the crepe twist in the spinning of the crepe yarn of Bemberg?

Mr. Benzing: Yes.

Mr. Beauregard: The question of boil-off has been brought out. Is there any distinction made between the effects of boil-off between soaps and the sulfated fatty alcohols, as far as the Bemberg Company is concerned?

Mr. Benzing: You mean as far as which is the better of the two is concerned?

Mr. Beauregard: That is right.

Mr. Benzing: We don't advocate anything in particular.

Mr. Beauregard: That has not been determined to your satisfaction—the beneficial effects of either operation?

Mr. Benzing: No. We don't recommend either.

MEETING, N. C. STATE COLLEGE STUDENT SECTION

THE North Carolina State College Student Section had as its guest on Friday, Oct. 3, 1936, Robert B. Murdoch, a graduate, now connected with the National Aniline & Chemical Co. Mr. Murdoch spent the day lecturing and demonstrating dye application.

Respectfully submitted, J. W. FURR, *Secretary*.

MEETING, NEW YORK SECTION

THE second regular monthly meeting of the New York Section was held on Friday evening, November 20th, at the Chemists' Club, New York City. The usual informal dinner was held at 6:30 P. M. preceding the meeting.

A very interesting and instructive motion picture entitled "The Story of Wool and Mohair," prepared by the Ford Motor Car Company, was presented by Mr. J. Walker of Sandoz Chemical Works.

Mr. Werner Von Bergen of Julius Forstmann & Company presented a paper on "The Dyeing of Wool Versus Mohair." The usual open discussion followed immediately after the presentation of his paper.

Dr. J. E. Meili, Chairman of the Program Committee, intimated we may look forward to a most interesting speaker for our next meeting.

Mr. John J. Sokolinski, Treasurer, submitted a financial report.

Mr. George L. Baxter, Chairman, spoke for a few minutes regarding the next annual meeting to beld at Providence in December.

A motion was offered and seconded, suggesting that each member and guest be furnished with a badge showing the individual's name for identification at the meetings. Mr. Baxter, Chairman, agreed to form a committee to give this matter immediate attention.

There was a total attendance of one hundred and ten (110).

Respectfully submitted,

P. J. KENNEDY,
Secretary.

UNEMPLOYMENT REGISTER

Members of this association, who are without employment, should forward to the secretary a statement as to their qualifications and experience. This information will subsequently appear on these pages with an identifying key number. Employers are also requested to file with the secretary or the American Dyestuff Reporter any vacancies which may occur in their businesses.—H. C. Chapin, Secretary, Lowell Textile Institute, Lowell, Mass.

Key: A—Laboratory and research; B—Dyeing or printing; C—Finishing; D—Sales; E—Instruction; F—Executive.

NOTE: PERSONS WHO HAVE OBTAINED EMPLOYMENT SHOULD NOTIFY THE SECRETARY OR THE AMERICAN DYESTUFF REPORTER AT ONCE.

Prospective employers may file vacancies either with the secretary or the American Dyestuff Reporter.

A-B-1

Education—Graduate chemist and colorist from a leading textile institute. Speaks French fluently. High standing at school. Awarded 1st prize in chemistry. Member of honor society.

Experience—Knowledge of most processes of textile manufacturing and some experience in woolen and worsted piece and stock dyeing and color matching. Presently employed in other department but wishes to get into laboratory or dyehouse.

A-B-5

Education—Graduate of the University of Lausanne, Switzerland. Doctor's degree in Chemistry. Knowledge of French and German.

Experience—Eleven years with large bleaching, dyeing, printing and finishing plant as assistant Chemist and then Chemist in charge of the Laboratory. Experience in dyeing and printing cotton and rayon fabrics; research and analytical work. Woman.

A-B-7

Education—Evening school in textile dyeing.

Experience—2 years laboratory in dyehouse; 9 years dyer and foreman on hosiery, ribbons and rayon; 2 years assistant to superintendent. Can handle help efficiently. Desires position as supervisor or demonstrator of dyestuffs. Age 30; married. Prefers vicinity of New York.

A-B-C-1

Education—B. S. in Chemistry, Massachusetts Institute of Technology.

Experience—7 years with a prominent worsted mill as assistant chief chemist, chief chemist, and superintendent of dyeing and wet finishing. Thoroughly familiar with chemical problems of worsted industry. Age 33; single. Location unimportant.

A-B-C-2

Education—Graduate of Lewis Institute of Engineering of Chicago and graduate of Philadelphia Textile School (Chemistry and Dyeing department).

Experience—Has had extensive experience as assistant chemist and colorist in silk printing, dyeing and finishing; also dyeing raw stock wool; and dyeing and finishing cotton and rayon. Age 30; married; references.

B-3

Experience—Foreman piece dyer for the past 21 years. Can dye the following: celanese, rayon, cotton backs, wool, silk and mixed fabrics. Has knowledge of finishing and laboratory work.

B-F-1

Experience—As executive had charge of the dyeing of ribbons and piece goods for 6 years; spun silk yarn, fast and resisted dyeing for 8 years; hosiery for 12 years. Has been quite successful in all three of these mills. Age 50 married. Will go anywhere.

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MORE ON GETTING ALONG WITH PEOPLE—II

Theory and Practice

WE previously tried to show by fairly specific examples that there is a great deal to this subject of getting along with other people. Now we are well into a somewhat more difficult matter, the more general discussion of the problem or, in a sense, the theory of the subject.

It has to do with the characteristics of people and with the almost hopeless job of changing their ways. It is easy enough to sit around and criticize everybody else, but aside from that, as you observe people and think about what they do, you inevitably come to the conclusion that most people do not want to know anything new beyond a few little things that they may pick up in their own way; and that they do not want to change their ways of doing anything.

We are all very much like that. Some of us may be able to learn from books or experience and actually change our methods but very few are willing to be told by others. Strange as it is, most of us who think we have brains or originality are that way. Whether these remarks apply to you or to the people you work with, that attitude is a serious handicap to accomplishing things. Any one who can recognize it in himself should strive to overcome it. He will probably have to use strategy to get around it in other people. One needs a sense of humor because he is apt to find himself showing the same traits which, in other people, cause him so much trouble. While we may speak of rules, the fact really is that there are none. It is worth more to study the behavior of people, to see the various possibilities and to follow them through to any resulting action than to try to learn any set of rules.

You cannot have in mind all of the time all of the ideas and suggestions that might be made. Just let the fact register that they are important, or if you are skeptical, study into it by actual observation and see for yourself. Then, observe yourself and fellow men, see the needless friction, and think out more tactful ways of accomplishing results.

As far as the present dissertations are concerned you may have better luck if you do the opposite of some of the things suggested. Such action may fit best with your conditions, so do not take these ideas too specifically. Our purpose is really to call attention to the importance of a new way of thinking in your relations with other people rather than to try to tell you just what to do. Putting it in terms of the laboratory—your own experimental observations will be better in the long run than any other person's theory because they fit you and your conditions.

Your Own Character

Some people do not seem to have any trouble getting along with anybody; others cannot get along with anybody. Probably no one falls quite within these rather exaggerated states but we are all somewhere between them and most of us subject to some improvement.

Most of the attributes of human character come into the art of getting along with other people. Here are a few of the more important—and being friendly and agreeable is naturally one of them.

But, the real good fellow is not the bright young glad-hander who greets you with a cheery good morning, quite forgetting that he has already done it once or twice before on that same day. Appearances to the contrary, some of the cooler, more silent individuals really care more for people in general than do many who put on a much better show.

Be a good listener. This does not mean sit around all day and let somebody else talk. Turn the conversation where you want it to go and then pay attention to what is said. Remember that there is nothing more aggravating than to talk to a person who is not listening but is trying to think up something to say to "go you one better." Of course you may find it expedient to listen to some useless talk and, if so, it is best to really listen.

It is best, also, to be rather ordinary and not to appear too wise or important. There is no sense in trying to hide necessary knowledge that you have; it is well to say enough to show clearly that you know what you are talking about; but it is better not to show off much more than necessary. In general, others do not like superior people. Be as superior as possible but keep it to yourself except where a display of real knowledge will do some good.

Do not look lazy, even if it is necessary at some time to be doing nothing. There is no harm in being frankly idle if there is a reason for it, but any superintendent or manager can recognize a sudden scramble to appear busy—he has done it himself at some time or other.

Don't be one of the silly folk who would rather carry their point or have their own way than to be correct. There may be exceptions but that sort of thing usually will not get you anywhere. Mill and laboratory work depend upon facts rather than upon out-talking other people. Better spend the time trying to find out what the facts are.

We did not set out to give a dissertation on the moralities but getting along with people does depend upon such things as honesty, which should not be too flexible. Some men in business seem to have the queer idea that it is all right to have their employes lie for them while overlooking the very apparent probability that one who will lie for another person will be equally likely to lie to him. I do not happen to know any good reasons for lying about technical matters because they are usually matters of fact. Things either will work or they will not; they are true or they are not. They are good or they are not good. There are plenty of tests to cover questions of quality, and cost figures for checks on processes. Certain amounts of materials are required and certain results obtained. A little optimistic exaggeration may have a use at times, but you can't kid the laws of nature—very much.

Also, the fact remains that lies and silly excuses do not fool people as often as the tellers may think. I have let many of them pass, but put them on mental record as real evidence on the value of the person who sprung them on me. It may not always be wise to call a lie or a bluff. Watch for the next one and you can soon learn whom to trust and who should be dropped. Some people are very good at showing themselves up.

Much of the point of these remarks is that these things work both ways. The kinds of observations that tell you about other people will also tell them about you. We are all more or less alike and we all do some of the "bad" things that we criticize in others. The trick is to recognize your own bad points and live them down, and to recognize the lack of any good point and to try to cultivate it and get it into use.

Knitted Wool Underwear Fabric

(Continued from page 694)

ment on the wool is due to the action of chlorine and there seems to be no reason why chlorine gas itself should be less harmful than its aqueous solution. Moreover, from a general viewpoint, it does not seem likely that the application of chlorine gas to wool materials could be so accurately controlled as the application of a hypochlorite solution.

It is evident that further research is required on the unshrinkable finish to find a better method than the one now available.

The unshrinkable finish, using an acidified solution of sodium hypochlorite, is carried out thus. Firstly, a cold solution containing 10 lbs of sodium hypochlorite (15% available chlorine) per 100 gallons is prepared for 60 lbs of fabric. Then to this is added 5 lbs of hydrochloric acid of 28° Tw. or 2 lbs of sulfuric acid. The mixture is rapidly stirred and then the wool fabric is entered and well worked about in the liquor for about one half hour. The action of the hypochlorite on the wool is very rapid and for this reason it is essential that the entering of the fabric be as quick as possible and that no time be lost in distributing it evenly throughout the liquor so as to ensure an even treatment. The liquor should smell of chlorine.

After this treatment the wool fabric should be thoroughly washed with cold water and then treated with a dilute solution of sodium bisulfite for the purpose of destroying any residual chlorine in the wool; this point can be determined by testing the fabric with starch-iodide paper. Then follows a further thorough wash and the fabric is then ready for bleaching either by the stoving or the hydrogen peroxide process.

It should be noted that the sodium hypochlorite solution used in the unshrinkable process must be acidified. Curiously enough, a neutral or alkaline liquor is not effective. In fact an alkaline liquor attacks the wool seriously and gives it a distinctly yellow color which cannot be removed by bleaching with hydrogen peroxide.

As regards bleaching, much the better result is obtainable with hydrogen peroxide; the resulting white color is permanent even after much washing. On the other hand, stoving (that is, hanging the wool fabric in a chamber containing the fumes of burning sulfur) has long been practiced as a cheap and efficient method of securing a good white, but owing to the fact that it is a reduction process (the bleaching is produced by the action of moisture and sulfur dioxide gas) in contrast to the oxidation process with hydrogen peroxide, the white is not permanent and gradually yellows. Peroxide is more expensive as regards chemicals.

When bleaching by the stoving method, the wool fabric (after scouring and whilst wet and containing a certain amount of alkalinity from the soap) is hung from poles in a wooden chamber. The doors are then

closed and a pot containing a few pounds of roll sulfur is placed in one corner and ignited by application of a hot iron poker. The sulfur burns and fills the chamber with sulfur dioxide and this then commences to bleach the wool. Generally it is found most suitable to commence the process in the evening and allow the goods to remain until next morning. The fabric is then taken down and given a good wash in water.

There are several difficulties which occur in stoving. Firstly, if the sulfur does not burn correctly a large amount of sulfur is volatilized as such and a fine yellow film settles in patches on the wool fabric which is difficult to remove. This may be avoided by arranging that air can enter the bleaching chamber at the point where the sulfur pot is placed; the incoming air enables the sulfur to burn completely. Another difficulty is that pole marks sometimes occur; this appears to be due to absorption of moisture from the fabric by the wooden poles generally used. The trouble may be avoided by using wood poles made non-absorbent by coating or impregnation with a Bakelite resin. Any iron stains which may be in the fabric before hanging in the chamber can cause trouble by assisting in the formation of brown stains which are extremely difficult to remove. Another trouble is that of drips from the roof on to the fabric. Of course, all these difficulties can be overcome by taking appropriate precautions but constant vigilance is required.

In the hydrogen peroxide bleaching process the treatment consists essentially of steeping the fabric in a solution of hydrogen peroxide made slightly alkaline by the addition of a substance such as sodium silicate, ammonia, or trisodium phosphate, and rendered more efficient as regards its bleaching action by the further addition of a substance similar to sodium metaphosphate or pyrophosphate. After this, the fabric is thoroughly washed and dried.

Hydrogen peroxide bleaching is not quite so simple a process as at first appears, and indeed the process itself is very imperfectly understood from the chemical viewpoint. In the first place it is essential that as little hydrogen peroxide shall be used consistent with obtaining a good white color in the wool, and in the second, it is necessary that the white shall be entirely satisfactory. It is possible to bleach under different conditions and secure a better white in one method than in another using maybe two or three times as much hydrogen peroxide; this will indicate the wide limits to the efficiency of bleaching which is possible.

A safe method of bleaching is that of using a 3-volume hydrogen peroxide bleaching liquor made just alkaline to litmus (pH = 7.5) by the addition of sodium silicate. This invariably gives good results at a reasonably low cost and consumption of hydrogen peroxide. But to those who wish to secure a higher efficiency there is a process in which the bath is made alkaline as before to litmus with sodium silicate and then a further addition of about 1½ pounds of sodium metaphosphate or sodium pyrophosphate is made per 100 gallons of liquor. It is claimed that with these special additions a good white can be secured on the wool with less than the usual consumption of hydrogen peroxide.

One important distinction between the stoving and hydrogen peroxide bleaching processes must be noted; it is connected with the whitening of wool which has been chlorinated. When this chlorination process for the unshrinkable finish is overdone the wool acquires a yellow color especially when it is in contact with alkalis. The higher the degree of chlorination the deeper is the yellow tone. Now bleaching with sulfur dioxide is more effective in removing this color than is hydrogen peroxide bleaching. In any case it is generally impossible to remove the yellow tone completely but it will be found that the best result is to be obtained by stoving. In seeking an explanation of this difference it must be remembered that hydrogen peroxide bleaching is carried out under alkaline conditions whilst stoving is an acid bleach. This, taken in conjunction with the fact that the yellow tone is deepened by alkaline conditions, is probably a clue to the required explanation.

In some instances where the fabric is required to be finished a so-called natural shade, bleaching by stoving or hydrogen peroxide is liable to produce too good a white. In these circumstances, use can be made of the bleaching action of a solution of sodium bisulfite. The wool material is steeped for about one-half hour in a warm solution of sodium bisulfite and then thoroughly rinsed and dried. It is essential to wash thoroughly since if much bisulfite is left in the fabric the finished fabric has a 'sulfur' smell which is objectionable. As a matter of experience the removal of this odor is a matter of some difficulty if the bisulfite liquor is once dried into the wool. Hence the advisability of thorough washing before drying.

The chlorinated and bleached wool fabric is now ready for finishing and here one or two important points must be considered. As previously mentioned, underwear fabrics must be soft and silky in handle. In general, chlorination tends to make the wool harsh and it is only by stopping the process before the wool has been made completely unshrinkable that the wool can be left with sufficient softness. The peroxide bleaching of chlorinated wool can also still further increase this harshness. But the stoving method of bleaching tends to leave the wool softer. Thus just before finishing, the fabric must be carefully examined from the viewpoint of handle. If it is harsh then steps must be taken to make it softer.

In spite of the large number of proprietary agents which are being marketed for softening all fibers including wool it is not easy to soften wool which has been harshened by the unshrinkable finish. Strangely enough, an overchlorinated wool will have a high degree of softness so long as it is wet; it is when the wool is dried that it becomes harsh. The disadvantage of many of the softening agents advertised as being satisfactory is that they leave the wool

with a scroop. Chlorinated wool easily acquires a scroopy handle when treated with soap products.

In actual large scale practice the finisher finds it difficult to soften chlorinated wool materials because two opposing factors come into play. Firstly, in order to avoid the production of a scroopy handle in using soap softeners he is obliged to leave the wool somewhat alkaline; scroop is more likely to develop as the wool becomes acid. Secondly, as the wool is made more and more alkaline so does its whiteness change to a yellower tone; from the viewpoint of color it would be better to leave the wool acid. Thus the finisher has to compromise and this compromise must be determined by circumstances. The finisher can best tackle this problem by bringing the fabric always to a certain pH which he has found by experiment to give the best handle taken in conjunction with the color. It is likely that the most suitable pH will be 7.5 to 8.0.

When a super-softness associated with a full handle is required then it is likely that the fabric should be slightly raised so that its inner surface (that is, the inner surface of the garment) is slightly hairy. This can be readily carried out on a brushing machine provided with wire carded rollers. Then, on pressing, the hairy surface will be flattened and so give solidity to the fabric.

Whether or not the fabric is raised it must be finally pressed by means of a hot calendar or stationary press. In this process its surface becomes smooth and silky, whilst all creases are removed. Subsequently the fabric is conditioned whereby absorption of moisture and cooling softens its handle. The fabric is then ready for cutting up into garments or for despatch to the garment manufacturer.

It will be evident from the foregoing description of processing that wool underwear fabric requires much attention if a satisfactory finish is to be obtained. The individual processes must be carefully correlated with each other. Thus the finishing methods will be different according as the stoving or hydrogen peroxide bleaching processes are used; they will also have to be modified if the chlorination is omitted. This is because the handle of wool is very easily affected by comparatively trivial variations in its treatment — much more so than is cotton or rayon. Experience with this underwear wool fabric shows that the best system of dealing with it, is that of devising a sequence of treatments which give a good result and then keeping to this sequence as rigidly as possible.

TECHNICAL NOTES FROM FOREIGN SOURCES

Vat Dyeings Upon Wool

Dr. Fritz Mayer—Monats. f. Textil-Ind. 50, 41 (1935). —According to the group-characteristics of a vat dyestuff, it must be of itself insoluble in water, capable of being reduced to a form which is soluble in an alkaline bath, this solution possessing a definite affinity for a given fiber, and this alkaline salt of the reduced dyestuff, thus taken up by the fiber, being capable of subsequent reoxidation, in the fiber, to the original insoluble form by ordinary oxidizing agents, by air, at least, though such an agent as nitrous acid may often be used to accelerate the reoxidation. The dyestuff (really pigment) must also show at least a certain good fastness, when thus regenerated upon the fiber, and there must be no part of the process which will involve any damage to the fiber.

As to these definite requirements, the first, the capability of being "vatted," may be dismissed briefly as a familiar matter. As to the second point, the affinity of the reduced dyestuff for the fiber, our present chemical or physical knowledge does not enable us to make any prediction in a case still untested. We do not know why one vatted dyestuff has a high affinity for cotton, while a closely related one has a poor affinity, or in some cases no affinity at all. We do know a few means of producing or of increasing this necessary affinity, but we cannot express these means as equations, or indeed even lay down any general principles regarding the matter, although, e.g., we do know that a large increase in the number of CO groups often diminishes the affinity.

The third point, that of ease of regeneration of the dyestuff when once upon the fiber in its vatted form, offers no difficulty. If the reoxidation in air is not satisfactory for any reason, the use of one or another oxidizing agent will overcome the difficulty.

As to the fastness of the dyeing when finished, we are in a position to make certain definite statements, since fastness in general is related to the susceptibility of the dyestuff to attack by various definite agencies — light-fastness as connected with oxidative degradation, for example. Fastness to washing is to some extent related to solubility of the dyestuff in alkaline baths. And as to the last point mentioned, that of possible weakening of the fiber during the process as a whole, we are learning more and more about it, and are coming into the position of being able to predict probabilities in definitely limited cases.

But, now, when we consider the widespread use of, and excellent results from, the vat dyestuffs with cotton, considering the topic with the above essential points in mind, we must emphasize the fact that the insensitivity of cotton toward alkali has been of the greatest importance, and that, further, no substantive dyestuff has been able to compete with the vat dyestuffs, until the Naphthol AS series came into use; and that the principle underlying the success of the newer group of dyestuffs (pigments) is again the general one of the generation of an insoluble pigment within the fiber.

If we now bring the case of wool into consideration, we have to recall that wool is very sensitive to alkali, so that,

in many vat dyestuffs recommended for wool, we must work at least in very weakly alkaline baths, which will make the reduction of vatting of the dyestuff more difficult.

In regard to the second point above, that of affinity for the fiber, differences are to be noticed. With wool, the affinity is frequently less than is the case with cotton, which would seem rather surprising. For example, the indigo vat for wool has to be heated to 50°-60° C., while with cotton dyeing takes place well in the cold. There exists, therefore, some relationship between affinity and temperature. As to the conversion of the reduced dyestuff to its final form, apparently the nature of the fiber is not involved. Yet the nature of the fiber does exert a strong influence upon the fastness of the dyeing, when this is accomplished. The author gives a table covering the fastness-properties of Indigo, Indigo R, Ciba Blue B, and Ciba Red G, toward light, washing, and water, for both cotton and wool. The figures for water are about the same for both cotton and wool; for washing, they are in general slightly lower for cotton, while for light they are much worse, in general, for cotton than for wool. The same general behavior is true for Indanthrene Red-violet RH (Helindone Red 3B).

Outside of the already well-known sulfur dyestuffs, the vat colors on cotton meet a serious competitor only in the Naphthol AS group. With wool, the case is different. The acid azo colors have entrenched themselves firmly in the field. They possess a direct and powerful chemical affinity for wool, while only a comparatively small subgroup of them, the direct cotton colors, possess any affinity for cotton, and even this one not comparable in intensity with that of the acid azo colors as a whole homogeneous group for wool—the affinity with the direct cotton colors is a physical rather than a chemical one. In addition, the process of afterchroming acid azo dyeings upon wool has greatly increased the range and fastness properties of the group. So that, against this seriously competing group, the vat colors for wool have to face not only this possession of the field by another, and large and valuable, group, but also their own characteristic defect of limited choice as to tone, the need of working usually in strong alkaline solution (strong in any case as related to the sensitivity of the wool), and a greatly higher price.

Yet, in spite of all this, vat dyestuffs for wool are a matter to be reckoned with, because of their intense fastness, particularly for fabrics intended for uniforms, and more particularly, perhaps, military uniforms—where, for psychological reasons, perhaps, failure to maintain quality would have unfortunate results upon morale. And, with a fair number of the newer vat colors, one can with care work well at a comparatively low alkalinity. Careful control in dyeing makes the use of the vat colors perfectly feasible; and carefulness is a factor, and an important one, in any textile process.

Physico-chemical Methods for Determination of Damage to Wool

A. Itkina u. Plechen—Mell. Textil-Ber. 16, 106 (1935). —A previous section of this paper has already been commented upon in these columns.

The present section includes five tables covering results of the methods already taken up and discussed—determination of the extent of damage to wool by determination of the amount of Benzopurpurine 10B adsorbed under standard conditions, the adsorbed amount being determined colorimetrically by determining the amount unadsorbed and remaining in solution, the amount of dyestuff adsorbed by a wholly undamaged sample of wool being taken as zero (table 2); the Alwoerden reaction (action of chlorine water upon the wool fiber) applied in a quantitative manner by microscopic linear summation of the sections of a single fiber showing the bubbles, characteristic of the reaction, in a given length of fiber (using the ocular micrometer) (table 3); the degree of swelling in various liquids, measured dry and after swelling, with the ocular micrometer (table 4); a short paragraph upon the determination of loss of weight of the fiber after treatment with water and potassium carbonate solution (table 5); and, finally, a discussion of the determination of amino-nitrogen still present in damaged wool, again by the indirect method, that is, by determination of amino-nitrogen present in various solutions after undamaged wool has been treated degradatively in them—the amount of amino-nitrogen found in such solutions bearing some relation, at least, to the loss of such nitrogen by the fiber (table 6).

The two sections of the paper do not claim to have done more than call attention to the fact that exactly performed tests can and do give exact information as to the actual degree to which wool has suffered damage by various treatments or by plain carelessness. It is a laudable effort to show textile technicians that matters in question can be determined scientifically, and determined exactly, instead of being loosely tested and thereby made subjects of opinion only. The author's suggestions, if put into general practice, should gradually bring about the replacement of the interminable discussions about facts not carefully studied, which fill too large a space in our journals and public meetings, by shorter and definite statements about facts which have been determined and delimited.

The paper is really an invitation to workers to begin employing such exact and unimpeachable methods, so as to replace opinion by actual knowledge. It is a thoroughly good paper.

Papers presented before the Annual Meeting and stenographic reports of the discussion will appear in future issues of the Reporter.

Lecithine in the Textile Industry

H. Tatu—TIBA 12,427 (1934)—Lecithine is a naturally-occurring substance whose use has been strongly recommended, during recent years, as an assistant in various textile processes. The substance itself is classified as a fat as well as a phosphatide, according to whether more weight is laid upon the presence of two stearic acid residues, or the one atom of phosphorus organically combined, in the molecule. Its constitution, possibly, is to be regarded as that of a derivative of glycerophosphoric acid, into which a choline residue is esterified. It occurs in many animal and vegetable sources, usually in very small amounts; in particular, in egg-yolk, from which it was first extracted in any considerable quantity. Now-a-days, the principal source is the soja-bean, from which it is obtained by moistening the beans, powdering them finely, and extracting the lecithine by a suitable solvent the product being a mixture of an oil and lecithine, the latter being separated from the oil usually by a coagulative process. The commercial product is usually a yellowish, pasty mass, greasy to the touch, and dispersing excellently in water, forming a stable emulsion, which is very stable in the presence of alkali, as well as lime; it is destroyed (the emulsion) by acids, normal HCl splitting the lecithine into choline and the distearate of glycerophosphoric acid. The lecithine can be prepared in powder form also, without losing its emulsifying properties.

The purity of commercial samples is not high; about 60%, sometimes as low as 50%, rarely above 65%. Its analysis is tedious, and depends upon the determination of the phosphorus content by the gravimetric method (the phosphomolybdic method). The volumetric method, with uranium acetate, is less exact.

The value of the substance lies in its twofold possibilities, of acting as a finishing agent pure and simple, its solutions possessing excellent penetrating power and the finish being soft and supple, and of acting also as an excellent emulsifying agent. Its range in this direction is very great; dilute emulsions are very stable; but, in working with strong solutions, approximating the condition of pastes, the emulsion may suddenly pass from the type of oil-in-water to that of water-in-oil.

The Examination of Mildew on Cotton

WILLIAM G. CHACE*

IN 1925 Bright[1] first published his "Cotton Blue" method of differentiating mold on cotton. His method has been copied by many including the present author[2]. In this last named article it was pointed out that the method was not entirely satisfactory with the dye then used—C. I. 707. In view of the poor results, experiments have been carried on to determine which of the cotton blues (there are eight of them listed) was most satisfactory for this test.

As a result of these experiments it has been found out that if the test is carried out as described by Bright using a stain sold under the name of Poirier's Blue (National Aniline and Chemical Co.'s stain) that results exactly like those secured by Bright are obtained.

Conn[3] states that this stain is a mixture of undetermined constitution and not listed by the Colour Index.

The test is carried out as follows:

The sample is warmed in lacto-phenol on the slide 1 minute. It is then transferred to a 1% aqueous solution of Poirier's Blue and warmed 3 minutes. It is then washed in water and warmed in lacto-phenol to remove as much color as possible. The sample may be examined immediately in lacto-phenol or dried and mounted in thick Canada balsam. The cotton is only slightly colored, the mold is stained a deep blue.

Examination with an Eastman differential color filter with orange ring and blue center gives the best differentiation.

* Lowell Textile Institute.

[1] Bright, J. Roy. Mic. Soc., 141 (1925).

[2] Chace. Am. Dyestuff Reptr., 20:801 (1931)

[3] Conn. "Biological Stains," Geneva, N. Y. (1929).

TRADE NOTES — NEW PRODUCTS

• BOOK ON METAL PROBLEMS

A new booklet describing the solution to scores of actual metal problems as encountered by the engineer has just been issued by the International Nickel Company. Containing 48 pages, illustrated throughout, this booklet has been prepared primarily as a guide book to Monel and other non-ferrous nickel alloys in the fields of enginering applications. It also covers the corrosion resistance and other properties of these metals.

The booklet includes 20 sub-divisions, each devoted to specific problems in fields from hydro-electric and steam power plants to highway maintenance, refrigeration and automobiles.

Besides going into details of standard Monel the booklet describes some of the newer forms of this alloy, including "K" Monel, "S" Monel and the like. The non-magnetic forms of the metal are also covered in a special section devoted to airplanes and the like. Inconel, the high nickel-chromium alloy, is another subject covered.

Special items included are those dealing with meters and other regulating equipment, sewage disposal, pump maintenance and other items of general interest. The booklet is available without charge to members of all branches of the engineering profession.

• DU PONT RELEASE

The Dyestuffs Division of E. I. duPont deNemours & Co., Inc., announces release of the following new product:

Monastral Fast Blue B S*—recently introduced by the I. C. I. of Great Britain

* Trade Mark.

to the European market. By special arrangement with them the duPont company has obtained permission to use their name "Monastral." It is an insoluble pigment said to produce extremely brilliant shades of blue. It is manufactured in both a highly dispersed powder form as well as a paste. It is claimed to be particularly resistant to most of the severe color destroying agencies and because of its fastness properties and tinctorial strength, it is recommended by the manufacturers for use in the printing ink, lake, paint, wall-paper, paper, linoleum, cement, leathercloth, textile and other industries using color. It is said to be suitable as a throwster color in the manufacture of woolens and worsteds. For this purpose it is applied in the customary manner and is said to possess good fastness to light and to be easily scoured from the yarn.

• BURKART-SCHIER PLANT

The Burkart-Schier Chemical Co. announces the construction of a new warehouse and manufacturing plant which is located on 13th and Chestnut Streets in Chattanooga, Tenn. It is of brick and steel construction with concrete floor and containing 25,000 square feet of floor space. Part of the new building will be used for chemical storage and part for a sulfonating plant. The new building is connected with the old by covered runways and loading platforms. The two buildings will occupy the block between 12th and 13th Streets. General offices will be located on the second floor.

A modern plant control laboratory is located in the manufacturing division. Experimental and testing laboratories are equipped with Monel metal and stainless steel equipment. New nickel and stainless steel tanks have been installed in the manufacturing division. It is claimed that the sulfonated oil plant is one of the most modern in the country. The old plant will be used for acid and heavy chemical storage. The Burkart-Schier company manufactures sulfonated oils, softeners and specialties for the textile trade and act as jobbers of textile and industrial chemicals.

• WITE-BRASS

The Alrose Chemical Co. is now placing on the market a "white brass" plating solution under the trade name Wite-Brass. This is claimed to be as white as silver, to be non-tarnishing and to be extremely simple to control. This product was developed by the Metallurgical Division of the Alrose company. It is stated that this division has also developed the Jetal Black process for finishing iron and steel. The company was also a pioneer in Rhodium plating—said to be a highly important non-tarnishing finish for a wide variety of products such as jewelry, optical goods and reflectors.

• DR. GANSEL JOINS ONYX STAFF

To further extend its policy of cooperation and service to the processing trade, the Onyx Oil & Chemical Co., Jersey City, N. J., announces that Dr. Eugene Gansel has joined its staff as field consultant and demonstrator.

Dr. Gansel is well known throughout the trade for his work as "trouble-shooter" over a period of nine years with the United Piece Dye Works, Lodi, N. J., and for his successes in process development with that company.

Dr. Gansel will render practical mill assistance to dyers and finishers on any problems concerning dyeing, bleaching, boiling-off silks, scouring synthetics, printing and finishing.

• GENERAL RELEASES

General Dyestuff Corp. announces release of circulars on the following new products:

Direct Fast Orange 5 R A S—a new direct orange manufactured by General Aniline Works and offered to the trade by General Dyestuff. It is claimed that it has the principal advantage of drawing well on cotton even at a low temperature without staining animal fibers to any extent. It is therefore said to be especially suitable for covering the cotton in union dyeing, either near the boil or at a low temperature as in speck dyeing. Circular G-157.

Astra Cyanine B—a straight basic color said to be distinguished by exceptional brilliancy. Circular I.G. 1237.

Appretan S F—a non slip finish of I.G. manufacture for rayon fabrics. Fabrics treated are said to have a good handle and attractive appearance. It is recommended by the manufacturers for rayon piece goods such as linings, etc., to prevent the warp and weft threads from pulling apart. It is said to stick the warp and weft threads together with an elastic film at the same time acting as a filling agent. Circular I.G. 1229.

Leophen K—a mercerizing auxiliary of I.G. manufacture said to be not only an excellent wetting out agent for mercerizing liquors but to also have the property of not forming a consistent foam, which is important from a technical point of view in the mercerizing of pieces of yarn. It is a slightly viscous brown liquid which is said to dissolve to a clear solution in mercerizing liquors of 48 to 57° Tw. It is stated that the liquors do not precipitate on prolonged standing. Circulars I.G. 1244 and 1245.

Toluylene Yellow R L—a direct dyestuff said to be distinguished by very good dischargeability, good fastness to light and fairly good fastness to washing. Circular I.G. 1214.

Diazo Fast Yellow R R—which, when diazotized and developed with Developer Z A, is said to yield pure yellows of excellent dischargeability and good wash and light fastness. Circular I.G. 1218.

• STUDY OF PRODUCTION AND DISTRIBUTION

The study of trends in the organization of production and distribution in the textile industries which was announced in July is now well under way. This study is being made for the Textile Foundation by the Industrial Research Department of the Wharton School of Finance and Commerce of the University of Pennsylvania under the general direction of Dean Joseph H. Willits. The Committee on Economic Research of the United States Institute for Textile Research is sponsoring the study in cooperation with the presidents or managing directors of the following textile trade associations:

Association of Cotton Textile Merchants of New York, Cotton-Textile Institute, Inc., International Association of Garment Manufacturers, National Association of Finishers of Textile Fabrics, National Association of Hosiery Manufacturers, National Association of Wool Manufacturers, National Federation of

New Warehouse and Manufacturing Plant of Burkart-Schier Co.

Textiles, Inc., National Knitted Outerwear Association, National Rayon Weavers Association, Textile Fabrics Association, Underwear Institute and Wholesale Dry Goods Institute, Inc.

The inquiry is centering on those developments in organization which have involved on the one hand sellers endeavoring to achieve control over their markets, and on the other hand buyers attempting to achieve control over their sources of supply. For example, some weavers, have licensed users of their fabrics and others have gone so far as to establish their own cutting plants; and at the same time some retailers have established close contacts with garment factories and cloth mills, and cutters with weavers or converters. In other words, the study is being focused on those developments in textile production and merchandising which involve vertical integration, either formerly through ownership or informally through contractual arrangements. These developments are being appraised for what they may reveal about the factors which make for success or failure of vertical organization in textiles and the results obtained from such organization.

The report is being based upon an analysis of the experience of those companies both inside and outside of the textile industry which have tried some form of vertical organization or merchandising in the last ten to fifteen years, and will range from yarn producers in the case of rayon fabrics to retailers in the case of all except industrial goods. In analyzing company experience care is being taken to distinguish between those factors which appear to have industry-wide influence and those which seem peculiar to individual companies. As a background for this intensive study, some consideration is being given to the forces which appear to have generated the present set-up for producing and marketing textiles and textile products, and the extent to which these forces may affect vertical integration where it is now under way.

With the initiation of the study in July the Industrial Research Department began a series of interviews with individuals qualified to advise upon the general conduct of the study and upon the companies which ought to be included in the case analysis. Conferences were held first with the members of the Sponsoring Committee individually, followed by discussions with the presidents or managing directors of the twelve cooperating textile trade associations. These conferences were followed by interviews with a number of executives in the various textile industries who were known to have given considerable thought to the problems of production and distribution organization in their respective industries. In addition the study has been discussed with clothing manufacturers, retailers, factors, and others well informed on some phase of textile marketing or production. Much interest has been expressed in the study by those interviewed and all have evidenced a willingness to cooperate.

● STANDARDS ON TEXTILE MATERIALS

In order to make available in their latest approved form all of the 42 A.S.T.M. standards covering various types of textile products the A.S.T.M. has issued a revised and amplified edition of this publication which is sponsored by Committee D-13 on Textile Materials.

During the past year, nine outstanding technical papers were presented at meetings of the committee and extensive abstracts of these papers are given in the publication.

The 1936 edition contains for the first time new methods of testing applying to pile floor covering; fineness of wool; corded cotton gray goods; yarn slippage in silk, rayon and silk-rayon woven broad goods; and fastness to laundering or domestic washing of dyed or printed cotton fabrics and printed silk or rayon fabrics.

A section comprises many excellent photomicrographs of common textile fibers and a convenient yarn number conversion table. Also included are proposed methods covering the testing of wool felt and correction of breaking strength to standard regain.

● pH REDOX METER

The Series 3 Coleman Electrometers are claimed to be light weight, portable precision instruments for the determination of pH and Redox in field, factory or laboratory. Based on the same fundamental principles as the "Coleman pH Electrometer" these new developments are said to be more compact, more sensitive and very much simpler to operate. Sturdy mounts protect the unusually small sealed glass and reference electrodes against rough handling while allowing a convenient and highly accurate cell chain free from ground glass joints or fragile glass parts. It is stated that accurate determinations of either pH or Redox can be made with less than 3 ml of sample with the regular electrode assembly while special micro assemblies are also offered. Means are provided for compensating both temperature and assymetry potentials by calibrating against a known buffer. It is further said that because of the electronic amplifier micro redox electrodes cannot polarize, allowing routine oxidation-reduction tests under conditions usually considered impossible.

Model 3 Electrometer

Model 3A reads directly in pH; model 3C reads both pH and Millivolts, allowing the determination of Redox as well as pH.

Both instruments are offered complete with sealed electrodes, batteries, tubes and standardizing buffer.

Descriptive literature and information can be obtained by writing to the manufacturers, the Coleman Electric Co.

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POSITION WANTED: Dyer. six years' experience on cotton, woolen and worsted. Graduate chemist. Single, thirty years old. Desires position with sales organization or manufacturer. Write Box No. 934, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

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